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COOLING LIQUID CHARACTERIZATION AND TESTING METHODS

Mudura Pavel
University of Oradea, pmudura@uoradea.ro

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Abstract : This paper is a summary of methods used to characterize the cooling fluids used in quenching steel, we will outline the objectives and the working principles of testing and evaluation methods for these liquids.

1. INTRODUCTION

Among the current metal processing technologies, thermal treatment is of high importance, being responsible for the technological and final properties of the constructed part. The cooling procedure, a basic thermal treatment procedure, is crucial, and the way that these procedures are executed, influences the mechanical, physical, chemical, and technological characteristics of the thermally treated products (parts, tools, semi-products). This influence is due to the resulting modification of the structure and internal tension. The cooling procedure can be considered the the most sensitive and demanding operation.

The cooling procedure is carried out with the help of cooling mediums, which have a significantly lower temperature than that of the piece heated in advance. The main industrial cooling mediums can be classified as follows: liquid, gaseous, solid and mixed (gas+liquid, gas+pulverized solid particles).

In practice, there are two types of cooling methods: continuous and discontinuous.

In the case of cooling liquids- used in thermal treatment technologies, depending on the way in which the liquid comes into contact with the piece, the cooling can be done: by a jet, through immersion, or through pulverisation. Because of the diversity of these liquids, it is necessary to characterize them from the viewpoint of their current usage, especially from the viewpoint of the structures formed during quenching. Thus, it is necessary to define a size typical for the cooling process, allowing: classification, comparison, following the evolution of liquids, etc. In order to better meet these requirements, researchers have proposed several methods for testing and characterizing cooling liquids, using different types of samples to achieve the cooling task.

2. CHARACTERISTICS OF COOLING LIQUIDS.

In recent decades, different cooling mediums have been tested and used, with the scope of optimizing the quenching of steel. Currently, the liquids and gases are the most commonly used.

At present, though the optimal cooling conditions of steel, regarding its quenching, are not accurately known, the progress made in studying cooling liquids allows us to obtain the desired structures, and also significantly reduce the risk of substantial errors. For a liquid to be used on an industrial scale, regarding the quenching of steel, it needs to posess a number of features that can be classified as follows:

a) *Usage characteristics*, that consider some unwanted side effects, chich may arise during the practical use of cooling liquids. In this respect, we mention the following characteristics: risk of fire, toxicity, pollution, degree of adherence, the risk of corrosion, leaching (the ease of cleaning the parts), resistance to oxidation, thermal degradation, etc.

b) Cooling features, which refers to the ability of these liquids to extract heat from a heated part. Basically, these features are reflected during cooling of certain samples, over certain temperature intervals, either regarding the cooling curve of a point inside the sample (time-temperature variation- T=f(t)). Generally, this is characterized by three stages of cooling [1]: calefaction, cooking, conveciton.

- *calefaction*, is characterized by a vapour coating around the sample.

Calefaction slows down the cooling process, while it is an unstable phenomenon, therefore hard to control. Calefaction [2,3] is favored by the followin factors: increase in coolant temperature, increase of the smoothness of the part surface, insufficient agitation of the quenching bath, small surface/volume ratio of the part.

Factors thet shorten the duration of the calefaction: the presance of more than 5% ionic substance in the liquid (sodium chloride, potassium, lithium, etc.), an oxide layer deposited on the workpiece surface (this presents a critical thickness at which the calefaction is completely eliminated, and the cooling will be faster and more regulated), a more vigorous agitation of the liquid, non-vaporisable liquids.

The calefaction's instability increases as the fluid becomes cooler, and results in slightly reproducible cooling. Therefore it is desireable to control the calefaction in order to shorten its duration as much as possible, or to stabilize it. This can be done by strong agitation of the liquid or by depositing a well chosen thermal resistance on the surface of the piece.

On the other hand, calefaction is present exclusively when cooling in gaseous mediums.

- *boiling*: in this stage, the vapour coating is destroyed, and the liquid comes in contact with the hot sample, achieving the most intense heat exchange, due to the nucleic boiling of the liquid. The presence of salts on the hot metal surfaces causes immediate nucleation.

Agitating the sample in the coolant lead to breaking the vapour coating and to a faster transition to the boiling stage, resulting in an increased cooling rate in all three stages.

- *convection*, which begins as the sample temperature drops below the coolant's boiling temperature. In this stage, heat exchange occurs through conduction and convection in the coolant, and the cooling rate decreases substantially.

These three phases of sample cooling occur on the sample's surface, and they are reflected in the aspect of the cooling curve and the cooling speed curve corresponding to a point at a certain distance from the cooled surface.

Depending of the cooling liquid's nature and the technological parameters of cooling, the cooling curve T=f(t) may contain: three stages (calefaction, boiling, convection, in case of vapourisable liquids), two stages (boiling, convection, in case of strong agitation) or a single stage (convection, in case of non-vapourisable liquids)

c) *Quenching characteristics,* which refers to a specific liquid's ability to achieve quenching structures (martensite or/and bain) as deep as possible in a specific steel sample. These characteristics are reflected by the „U" curves, which represent the variation of hardness on the cross section of a cylindrical sample that was hardened through cooling, by immersing it into the cooling liquid.

The quenching characteristics are specific to each liquid, and are influenced by the thermokinetic properties of the liquid from the beginning of the cooling process: temperature, relative part-liquid speed (agitation), viscosity index (viscosity-temperature relation), density, etc.

The performance of a cooling liquid is determined by the characteristics mentioned above, and also by local conditions of use: type and size of the cooling system, the sample cooling system - sample heating system ratio, etc. To better highlight these performances, we need to establish some representative sizes, which can be determined using certain testing and evaluation methods of these liquids.

3. COOLING LIQUID TESTING AND EVALUATING METHODS.

3.1 OBJECTIVES

Testing and evaluation of cooling liquids concerns a series of objectives,that can be classified as follows:

a) *objectives concerning the supplier and user:*

- establishing a common language between supplier and user, and the criteria for choosing the coolant;
- presentation of the liquid coolant by the supplier, in a standardized form, in terms of their ability to harden steel.

b) objectives concerning the liquid coolant*:*

- characterizing the coolant's ability to provide the hardened steel with optimal properties.
- specific tests for the characterization and monitoring of the liquid coolant..
- determining the best balance between increasing the liquid's life to the maximum, and minimizing risk due to changes in performance during use.
- classification and comparison of cooling liquids, even of those of the same type, that come from different sources, and are not standardized after their quenching properties.
- optimization of adding more liquid coolant in order to increase performance or to restore the coolant.

c) *objectives regarding the industrial plant:*

- evaluating the heterogeneous quenching in a certain plant.
- classification of industial cooling plants in terms of quenching performance.
- determining specific tests for characterizing the global cooling capacity of an industrial quenching plant.

3.2 REQUIREMENTS

To achive the objectives above, the liquid coolant testing and evaluation methods need to meet the following requirements:

a) *Promptness,* which implies a time as short as possible, between taking the cooling liquid samples, and obtaining the results, which will be can be used immediately.

b) *Reproducibility,* which consists of obtaining the same results after performing a number of repeated attempts, when characterizing a certain cooling liquid. Thus the method needs to relate to stable phenomena, and has to use a standard liquid that allows the quality verification of samples, and calibration of the measuring line. For aging tests, reference samples of the liquid will be used.

c) *Sensitivity,* which represents the property of the testing method to respond to the slightest change of the cooling liquid's characteristics, that influence the quenching, such as: hardness, hardened depth, internal stresses, deformations;

d) *Representativeness,* which means that the test result depends only on the liquid's cooling ability (type of the liquid, temperature, pressure, sample-liquid relative speed, concentration, additives) and eliminate all factors related to the sample (material, shape, size, surface condition);

e) *Correlation*: the test results need to make the best correlation between the paramaters of the quenching results possible (hardness, hardened depth, internal stresses) and parameters defining the coolant (fluid type, temperature, agitation);

f) *Standardization of testing,* which involves the possibility to specify all sizes and operations that assure the reproducibility of test results, in case of a specified work hypothesis;

3.3 CLASSIFICATION

Lately, besides existing cooling liquids, a series of polimer-based cooling liquids have been inroduced, that are being used on an increasingly large scale. As a consiquence of the variaty of available coolants and the new liquids, arises the necessity to classify them. Thus, over time, a variety of coolant testing and evaluation methods have appeared. Given the components of the quenching process, these methods can be classified as follows: [4] :

- *thermophysical methods*, targeting the coolant's ability to extract heat from a metal sample while it is cooling.
- *metellurgical methods,* which aim to determine the liquid's capecity to harden a certain steel by quenching.
- *physico-chemical methods,* using physico-chemical properties of the coolants, in order to determine their ability to maintain their quenching capacity during work.

Thermophysical methods use to charactarize coolants, the notion of *cooling capacity*, while the metallurgical methods use the notion of *quenching capacity.* These two terms, accordint to „Metals Handbook" [5], are defined as follows: the *cooling capacity* is the thermal response of the coolant, or the mode of heat removal from a sample, usually standardized; the *quenching capacity* is the metalurgical response of a steel sample, or the liquid's capacity to create quenching structures in a given section of the steel.

A summary of the testing and evaluation methods of cooling liquids, is shown in in table 1.

3. CONCLUSIONS

Although various metheds have been developed for the testing and characterization of cooling liquids, none of these have yet been unanimously accepted. Existing methods can be grouped into two major categories: *methods for determining the cooling power of coolants (thermophysical methods)* and *methods for determining the quenching power of of coolants (metallurgical methods)*. Also, numerous attempts have been made to determine a link between the cooling and quenching power of a particular cooling liquid, resulting in the *cooling power to quenching power of coolants correlation methods.*

Table 1 Testing methods for quenching liquids

Method type	Method	Sample	Liquid	Determined parameters
1	2	3	4	5
Thermo-physical	Cooling curves method [1,6,7]	- „inconel 600” cylinder: ϕ12,5x60 - heated to 850°C	- unagitated oil: 2 l	- cooling time up to 600, 400, 200°C - maximum cooling speed - temperature at which maximum speed occurs - cooling speed from 300° C
		- silver cylinder ϕ16x48 - heated to 800° C	- unagitated oil: 700 ml	- transition temperature between calefaction and boiling: θ_1 - transition temperature between boiling and convection: θ_2
		- silver cylinder ϕ16x48 - heated to 800° C	- unagitated oil: 700 ml - aqueous solutions	- temperature θ_1 - temperature θ_2
		- silver cylinder ϕ10x30	- unagitated oil: 250 ml	- temperature θ_1 - cooling time from 800 to 400° C
		- silver ball ϕ 20	- oil	- cooling curve
		- silver cylinder ϕ8x24	- oil	- cooling curve
		- steel cylinder Stainless, with semispherical extremities ϕ70x120	- oil	- cooling curve
	O. Dorigo method	- soft steel ball: ϕ 50,8	- oil	$\Delta^o = \frac{44}{t_{300}^{800}}$; $44 = (t_{300}^{800})_{apa}$
	Houghton method [8]	- steel cylinder ϕ 55x50	- oil	- cooling time from 800 to 300°C
	Temperature gradient method [9]	- stainless steel cylinder ϕ 50x200	- quenching liquids	- curve: sample surface heat flux - time
	Hot wire test [10]	- wire ϕ 0,3, Ni-Cr	- oil	- current strength to break the wire
	Cooling interval test (calorimetric test)	- austenitic steel cylinder ϕ25x75 - heated to 845°C	- oil: 1800 cm^3	- oil temperature variation after 5, 10, 60, 90 sec.
	"5 sec." test[5]	- austenitic steel cylinder cca. 350g - heated to 815° C	- oil: 1800cm^3	100 A/B%
	Magnetic method	- nickel ball ϕ25 - heated to 835° C	- oil: 200cm^3	- cooling time from 835 to 355° C

Table 1 (continued)

1	2	3	4	5
Thermo-physical	Heat exchange coef. method, α [10]	Silver sphere Φ10mm	- test liquid	- cooling curve; - cooling speed curve;
	Relative heat exchange coef. method, H. [11]		- steady water - test liquid	- cooling curve of the reference medium (steady water); - cooling curve of de test liquid;
Metallur-gical	Jominy test [12]	- steel cylinder ϕ25x100	- test liquid	- Jominy curve
	Quenching by immersion method [13,14]	- steel cylinder sample	- test liquid	- "U" hardness curve
		- cylindrical sample in steel steps	- test liquid	- the sample section center's hardness, in different zones
		- wedge sample	- test liquid	- HV hardness at 3 mm from the edge
	Metoda CETIM [15]	- cylindrical steel bars: ϕ 20; 30; 40; 50; (L= 3D) heated to 850^{o}C	- 60l oil, unagitated to 50^{o}C or 150^{o}C	K = D / J
Physico-chemical	Test methods for oil products	–	- quenching oils	- color - density - flash point - point of combustion - viscosity - viscosity index - acidity index - foaming indice - ash content - Conradson residue - sulfur content - water content

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