

Deep Cold Treatment of Steel Parts and Tools

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Abstract: - It has been shown that the deep cold treatment of steel parts, especially made of tool steels, results in additional strengthening (superstrengthening) of material and essentially reduces the tool deterioration. The essential effect is reached if the deep cold treatment and austenite-to-martensite transformation coincide. This is explained by the creation of extremely high density of dislocations in the material, while these dislocations are frozen and do not accumulate at the boundaries of grains in the form of potential microcracks. The uniform distribution and dislocation freezing result in the release of nano-carbides. Long holding at the low temperature results in fastening (capturing) dislocations with atoms of carbon and other alloy elements. The high density of dislocations and their uniform distribution is the cause of high strength and plasticity of material and their wear resistance. To get stable results of cryogen treatment of steel parts, thermal and physical bases for the control of phase transformations have been developed, which include two-step cooling. At the first step the part is cooled to the martensite start temperature. At the second step, depending on the material, the part is intensively cooled within the martensite range or deeply cold-treated. This approach allows to obtain stable and reliable results.

Key Words: - two-step cooling, deep cold treatment, superstrengthening, wear improvement, tools

1 Introduction

The deep cold treatment in the beginning was applied for the transformation of the residual austenite into martensite and stabilization of tool sizes. Later it was discovered that there existed the phenomenon of the steel superstrengthening at high cooling rates within the martensite range [1]. It was noted also that the cooling tools to the temperature of liquid nitrogen increased its service life, in some cases by ten times [2]. The boom of cryogen treatment of tools by liquid nitrogen occurred in the former Soviet Union for many years: 1972-1980 [2,3]. However, the non-stability of results obtained and infrequent repeatability of positive effects dampened the ardor of researchers. At the present the interest in the deep cold treatment resumed and scientists got to deeply study at the modern level the effects obtained during such a treatment. Some of them that can be related to such studies are recently published papers [4,5]. The purpose of this article is to provide the foundation of phenomena observed and develop the appropriate technology and provide the stability and repeatability of the effects observed.

2 Two – Step Cooling

The two-step cooling with the delay or retention of martensite transformation at the first step is easily implemented using hot oils, different kinds of aqueous salt solutions and regularities of the self-regulated thermal process.

The duration of non-stationary nucleate boiling (self-regulated thermal process) is determined by the formula proposed by author [3]:

$$\tau = \left[\Omega + b \ln \frac{g_I}{g_{II}} \right] \frac{K}{a}, \quad (1)$$

$$g_I = \frac{1}{\beta} \left[\frac{2\lambda (g_0 - g_I)}{R} \right]^{0.3} \quad (2)$$

$$g_{II} = \frac{1}{\beta} [\alpha_{conv} (g_{II} + g_{uh})]^{0.3} \quad (3)$$

$$\beta = \frac{75\lambda'(\rho' - \rho'')^{0.5} g^{0.5}}{\sigma^{0.5}(\rho'' r^* \bar{W})^{0.7} Pr^{0.2}}; \quad (4)$$

$$\bar{W} = d_0 f, \quad (5)$$

Where $\Omega = 0,24k$, $k=1,2,3$ — correspondingly for plate-shaped, cylinder-shaped and ball-shaped bodies; $b=3.21$;
 K is the Kondratjev form coefficient (m^2);
 a is thermal diffusivity of a material (m^2/s);
 λ is thermal conductivity of the material ($W/m K$);
 λ' is thermal conductivity of the liquid ($W/m k$);
 $\mathcal{G}_0 = T_0 - T_s$; T_0 is the initial austenitizing temperature;
 T_s is saturation (boiling) temperature;
 R is the radius of the cylinder, ball, a half of width of a plate (m);
 a_{conv} is the convection heat transfer coefficient (W/m^2K);
 \mathcal{G}_{uh} is underheating of quenchant up to saturation temperature;
 ρ' is the liquid density (kg /m^3);
 ρ'' is the density of a part quenched (kg /m^3);
 g is the gravity (m/s^2);
 σ is the surface tension (N/m);
 r^* is the latent heat of evaporation (J/kg);
 Pr is the Prandtl number;
 \bar{W} is the average growth rate of steam bubbles;
 d_0 is a dispatch diameter of bubbles;
 f is a frequency of dispatch of steam bubbles (1/s).

It should be noted that

- The average growth rate of steam bubbles $\bar{W} = d_0 f$ for different quenchants is different.
- The value \bar{W} depends on physical properties of a liquid and its vapor, and it practically does not depend on a material of a wettable surface to be quenched, and is the characteristic value for this liquid and its vapor.
- The experiments have not shown any effect of changes in the thermal loading of the surface by 4-5 times upon the average value of \bar{W} . The only parameter depending on thermal loading is the quantity of bubbles per unit of surface area. Some results of fine

experiments of Tolubinsky for measuring the value of W'' are presented in Table 1 [6].

Table 1. Dispatch diameter and frequency of the dispatch of bubbles for water and aqueous solutions.

Quenchant	D_0 , mm	f, 1/s	W'' , mm/s
Water	2.5	62	155
25% solution of NaCl	2.4	64.5	155
29% solution of Na_2CO_3	2.4	65	156

In view of a large number of bubbles and high frequency of their dispatch, the heat transfer coefficient reaches high values during nucleate boiling. Nevertheless, the cooling rate of the surface to be quenched within the martensite range can be very low or even approach zero, when the self-regulated thermal process is established. If the boiling temperature of a quenchant coincides with the martensite start temperature M_s , there is a delay in the transformation of austenite into martensite up to the time of appearance of the convection.

If the boiling temperature of the quenchant in reference to the CCT or TTT diagram corresponds to 50% martensite, then no more than 50% of martensite can be formed during nucleate boiling. The second part of the martensitic transformation occurs at the second step of cooling.

The basic stages for the implementation of this technology are:

- Quenching is implemented in liquid media so that there is no full film boiling.
- The boiling temperature at CCT or TTT diagrams must correspond to approximately 50% martensite.
- When the nucleate boiling finishes, which time is determined by formula (1), the parts are unloaded from the quench bath and are kept in air until the temperature field on cross sections of parts is equalized.
- Afterwards, the steel parts are cooled in liquid nitrogen or cold gases

The improvement of mechanical and plastic properties of material after two-step quenching with

the use of intensively agitated liquid nitrogen at the second stage is presented in Table 2:

Table 2 Steel mechanical properties at step quenching (in high-concentrated solution CaCl₂ – liquid nitrogen) depending on holding time at the first stage (specimens of 5-mm diameter)

Method of cooling	Time of the first stage of cooling, s	Steel grade	Strength, MPa		%	
			R _m	R _{p0.2}	A	Z
Two-step quenching in 50% CaCl ₂ solution with transport into liquid nitrogen (vibromixing)	1	Y7A	1610	1570	7.9	31
		60C2A	1960	1740	4	22
	2	Y7A	1510	1450	6.1	22
		60C2A	1750	1630	7	51
	3	60C2A	1670	1590	7	33
		60C2A	1430	1260	8.2	26
Quenching in 50% CaCl ₂ solution to complete cooling	Until complete cooling	Y7A	1460	1370	7.6	22
		60C2A	1430	1260	8.2	26

Y7A=(0.65-0.74 C; 0.15-0.35 Si; 0.15 – 0.30 Mn; <0.15 Cr; <0.20 Ni; <0.20 Cu) in %

60C2A=(0.58-0.63 C; 1.6 – 2.0 Si; 0.6 – 0.9 Mn; <0.3 Cr; <0.25 Ni; <0.20 Cu) in %

Table 3 Comparative data on mechanical properties of specimens made of Y7A and 60C2A steels, treated in different ways

Steel quenching	Temperature, °C		Strength, MPa		%	
	Heating	Tempering	R _m	R _{p0.2}	A	Z
In oil MS-20 at 150°C	<u>800</u>	<u>360</u>	<u>1350</u>	<u>1270</u>	<u>4.8</u>	<u>29</u>
	860-880	460-480	1580	1410	7	38
In 10% CaCl ₂ solution	<u>800</u>	<u>360</u>	<u>1440</u>	<u>1400</u>	<u>7</u>	<u>26</u>
	870	460-480	1420	1260	8.2	36
In 10% CaCl ₂ solution for 1 s with the transfer to still liquid nitrogen	<u>800</u>	<u>360</u>	<u>1450</u>	<u>1400</u>	<u>6.4</u>	<u>29</u>
	870	460-480	1460	1380	6.7	19
In 50% CaCl ₂ solution for 1 s with transfer to liquid nitrogen (vibromixing)	<u>820</u>	<u>360</u>	<u>1610</u>	<u>1570</u>	<u>7.9</u>	<u>31</u>
	880	460-480	1920	1740	4	26

Note: above line is for Y7A steel, below line is for 60C2A steel.

The author of Ref. [5] presents results of increasing the service life of tools due to reducing its deterioration (see Table 4).

Table 4. Field trial of wear improvements in deep cold treated tools

Tool Type	Tool Material (AISI No)	Improvement in wear rate (%)
Stamping die	D-2	1000
Punch	M-7	600
End mill	M-42	450
Drills	M-42, M-7, C-2	300
Milling cutters	M-7	250
Drill	M-42	200
Punch	M-2	100

3 Discussion

The analysis of studies made shows that the significant effect of tool strengthening and service life increasing takes place when the martensite transformation and cold treatment coincide. The use of the regularities of the self-regulated process gave the opportunity to conduct very subtle and clear studies. Two-step quenching was performed. At the first step the delay or retention of the austenite-to-martensite transformation was observed due to the presence of the self-regulated thermal process. At the second step the samples were cooled in intensively agitated liquid nitrogen. When at the first step the cooling was longer than the self-regulated process, the martensite transformations actually ended at the

first step of cooling and no additional strengthening (superstrengthening) was observed (see Table 2). It has been also established that for every steel grade there exists the critical cooling rate within the martensite range outside of which the additional strengthening of material is observed. The higher the content of carbon and alloy elements in steel, the lower the critical cooling rate. For this reason, the effect is more frequently and clearly observed in case of high-alloy steel quenching (see Table 4). The coincidence of martensite transformation and cold treatment results in the phenomenon that the appearing martensite plates deform the supercooled austenite and create very high density of dislocations, which are frozen at low temperatures (see Fig. 1). With the increase in carbon content of steel the specific volume of martensite increases, therefore plates deform the supercooled austenite stronger and the phenomenon of superstrengthening is naturally increases too. This has been confirmed by experiments with D2 steel, which carbon content reached 1.5%. The frozen uniformly distributed dislocations are fastened (captured) by carbon atoms and result in the appearance of nano-carbides. In our opinion, the cause of increasing the wear is two factors: the high density of uniformly distributed dislocations and nano-carbides. This issue is to be further studied deeper and in more detail.

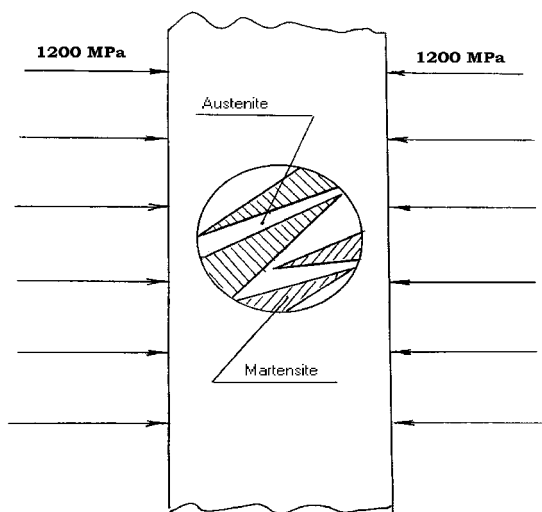


Fig.1 Scheme for the explanation of the mechanism of the material additional strengthening (superstrengthening).

4 Summary

- 1 Two-step quenching with the delay or retention of austenite-to-martensite transformations at the first step and deep cold treatment at the second step results in additional strengthening (superstrengthening) of material and increases its wear resistance.
- 2 Optimized ways of two-step quenching have been developed, which are based on the control of processes of phase transformations of austenite into martensite. They are based on the self-regulated thermal process, after which the deep cold treatment is performed.
- 3 The phenomenon of material superstrengthening and high wear resistance takes place due to the extremely high density of dislocations, which are frozen and uniformly distributed in the material and release of nano-carbides.

References:

- [1] N.I.Kobasko, Effect of Structural and Thermal Stresses on Crack Formation during Steel Quenching, All-Union Conference on Increasing the Heating Furnace Performance and Economy, Dnepropetrovsk, 1967, pp.26-27
- [2] E.S.Zhmd, USSR Inventor's Certificate 485161. Method of Heat Treatment of Tools, *Bulletin of Inventions* No. 35, 1975
- [3] N.I. Kobasko, *Steel Quenching in Liquid Media Under Pressure*, (in Russian), Naukova Dumka, Kiev, 1980, 206 p.
- [4] N.I. Kobasko, Steel Superstrengthening Phenomenon, *Journal of ASTM*, February 2005, Vol.2, # 2, Paper ID JAI 12824, available on-line at www.astm.org
- [5] P.F. Stratton, Process optimization for deep cold treatment of tool steels, *Proc. of the 1st Int. Conference on Heat Treatment and Surface Engineering of Tools and Dies (IFHTSE 2005)*, Pula, Croatia, 08-11 June, 2005.
- [6] V.I.Tolubinsky, *Heat Transfer at Boiling*, Kyiv, Naukova Dumka, 1980, 316p