# OPTIMUM PROCEDURES FOR VACUUM HEAT TREATMENT OF HOT WORK STEELS

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## ABSTRACT

The heat treatment of hot work steels is vacuum heat treatment application which requires a special attention. The final mechanical properties are influenced by many parameters related to the steel selection, manufacturing process, heat treatment and in special cases to the surface modification. The inseparable parts of problem are testing methods. The paper deals with principles which should be used in standard praxis which non-compliance results in low tool life or legal problems in supplier-customer relationships.

## 1. Introduction

Heat treatment of hot work steels is one of the most demanding disciplines in the field. Not only in terms of procedures, but especially in terms of determining and adhering to process parameters to achieve optimal life results with marked variability of heat-treated parts. However, when choosing process parameters, it is always necessary to take into account what procedures are ordered and what procedures are usual.

And here there is a discrepancy between the tool manufacturer's requirement to ensure tool life, and the procedure ordered or usual. It is a paradox that the customer does not usually have the usual procedures, or has no knowledge of them, on the other hand, this is exactly what he requires from the heat treatment supplier.

Of course, the next comprehensive chapter is the design of the tool, the choice of material and its hardness, or how to use the tool in production, but in this article we will assume that the customer has already investigated and brought this into line with the usual procedures "the best praxis" .)

## 2. Input material and its control

Although the group of hot work steels is extensive, essentially the same entry control rules apply to all steels. Its definition is contained in Nadca 207, rev. 2008 [1]. Although this definition is intended primarily for die casting dies, it can also be generally applied to other applications such as die forging, tools for extrusion of Al profiles, rolling or hot pressing.

| NADCA Grade | Material Category | C | Mn | P | S | Si | Cr | Mo | V |
|---|---|---|---|---|---|---|---|---|---|
| "Grade A" | Type H13 – Premium | 0.37-0.42 | 0.20-0.50 | 0.025 max | 0.005 max | 0.80-1.20 | 5.00-5.50 | 1.20-1.75 | 0.80-1.20 |
| "Grade B" | Type H13 – Superior | 0.37-0.42 | 0.20-0.50 | 0.015 max | 0.003 max | 0.80-1.20 | 5.00-5.50 | 1.20-1.75 | 0.80-1.00 |
| "Grade C" | Type 2367 & Modified | 0.32-0.40 | 0.10-0.60 | 0.020 max | 0.005 max | 0.10-0.50 | 4.70-5.30 | 2.00-3.30 | 0.40-0.70 |
| "Grade D" | Type H11/2343 | 0.35-0.42 | 0.20-0.60 | 0.025 max | 0.005 max | 0.80-1.20 | 5.00-5.50 | 1.10-1.60 | 0.30-0.60 |
| "Grade E" | Type H11 Modified | 0.33-0.43 | 0.10-0.70 | 0.020 max | 0.003 max | 0.10-0.55 | 4.70-5.70 | 1.10-2.10 | 0.30-0.80 |

***Tab. 1*** *– Classification of steels according to Nadca*

From the point of view of initial testing, the control of the chemical composition is a basic, but not a decisive condition.

The resulting mechanical properties are determined primarily by micropurity, grain size, amount and distribution of primary carbides. From this point of view, in order to distinguish precisely these influences, the usual procedure is to examine the input quality of the material by means of impact tests.

It is shown that the higher the impact strength of the material, the higher the resistance of the material to the formation of thermal fatigue cracks. A higher value of impact toughness can also positively affect the formation of macro cracks related to stress conditions.

Impact tests are performed on samples ideally hardened under oil cooling conditions and the measured values should reach the values defined by the Nadca 207, rev. 2008 (Tab. 3). In the event that the predicted values are not reached, it is the responsibility of the material supplier to replace the delivered material with a material that would provide the required parameters.

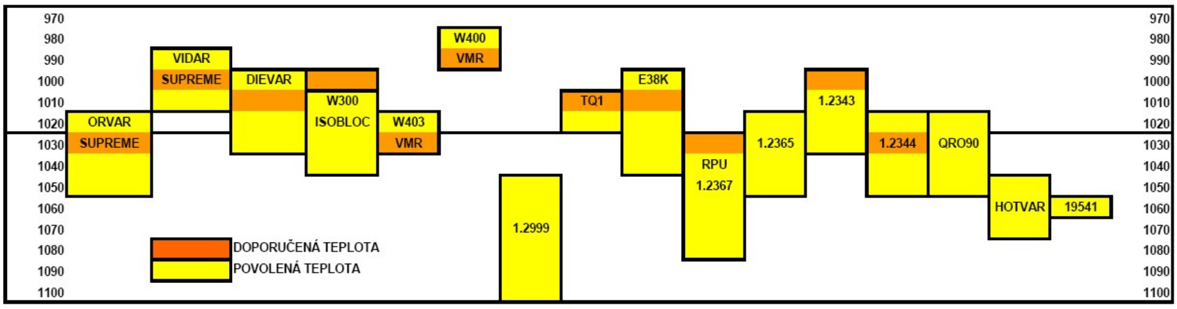
Impact tests are performed on samples with the least suitable fiber direction, thus ensuring that the ultimate mechanical properties of the material are evaluated.

## 3. Heat treatment

The heat treatment of these steels should be performed according to generally known, conventional procedures. It is a question of what these usual procedures mean. There are no Czech standards for most modern steels, there are only company material sheets. An exception is the standard Nadca 207, rev. 2008 [1], but it is only a recommendation for members of the die – casting association in the USA.

A properly managed heat treatment process should ensure that the expected mechanical properties according to the input material are achieved. Leaving aside possible problems with deformations during heating, then the most important phases of TZ are austenitization and cooling.

Austenitization should, on the one hand, ensure the correct dissolution of the carbon in the matrix, partial dissolution of the carbides, but on the other hand, it should not allow excessive enlargement of the austenitic grain compared to the initial state. These two requirements are in conflict and it is necessary to choose a compromise solution related to the time and temperature of austenitization, and with regard to the fact that we heat the 3D body in a heterogeneous environment of a vacuum furnace.




***Tab. 2*** *– The table of temperatures allowed or recommended for austenitizing*

If heat treatment according to the NADCA specification is not required, then the temperatures specified by the manufacturer of the steel used are considered to be the usual temperatures. If heat treatment is required by NADCA, then these temperatures are within the permitted range, usually at its lower limit (Tab. 2)

Although higher austenitization temperatures ensure better solubility of the primary carbides, these carbides, on the contrary, precipitate along the grain boundaries during cooling (hardening in a gas stream) and reduce the resulting toughness of the material. Paradoxically, due to the higher solubility of carbides, higher austenitization temperature means higher tempering resistance during the use of steel at the working temperature of the tool (Fig. 1), greater resistance to thermal fatigue cracks, but also a high risk of grain growth.

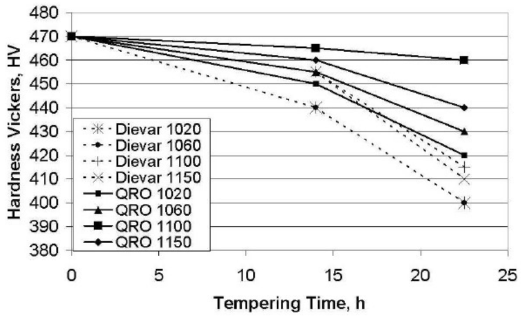
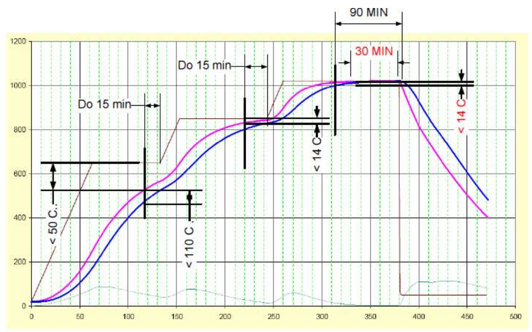



***Fig .1*** *– Temper resistance of quenched and tempered Dievar and QRO90, tempered on 600 C [2]*

In addition to the temperature and time of austenitization, the cooling rate is also important, which reduces or limits the possibility of precipitation of these carbides. The higher temperature also reduces **Ms** and increases the amount of residual austenite in the matrix due to the more carbon-enriched austenite. It then transforms into not-tempered martensite, or cementite and ferrite, and in all these cases the toughness decreases

The second most important parameter of austenitization is time. The delay at the austenitization temperature must be sufficient to homogenize the austenite, while the carbides will also partially dissolve, but must not allow excessive, uncontrollable growth of the austenitic grain. The higher the temperature the shorter the austenitization time should be. If the usual heat treatment is required, the steel manufacturers' instructions must be followed. In the case of heat treatment according to Nadca, then the austenitization time is the time prescribed as 30 minutes from the complete heating of the part to the end of the delay if the following conditions are met (Fig. 2):



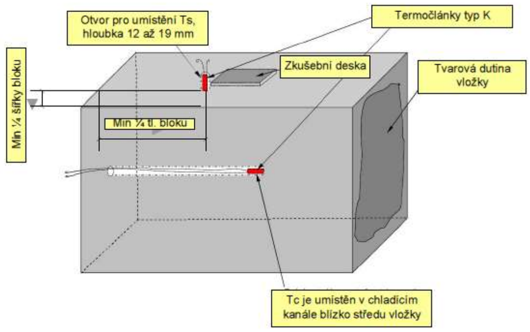




*Fig. 2 – Temperature control of vacuum furnace during the heating up and austenitizing*
*Fig. 3 – Location of thermocouples Ts(surface) and Tc(core)*

***Ts (surface) = Tp (programmed)*** and ***Ts (surface) -Tc (core) < 14 C***

If the furnace is not equipped with 2 load thermocouples Ts and Tc, the maximum holding time at the temperature is allowed to be 90 minutes from **Ts = Tp**

Therefore, in order for this parameter to be objective and controllable, the location of the thermocouples must be defined in addition to the above conditions. The tool manufacturer is responsible for making the correct holes for the **Ts** and **Tc** thermocouples. (Fig.3).

A very important parameter of the process control is the cooling rate in the area between the austenitization temperature and reaching a temperature of approx. 400-450 ° C. This should be a controlled parameter with a minimum value of **28 °C/min**. Only under this condition can the area of carbide precipitation in materials of types H11 (1.2343) and H13 (1.2344) be missed, which means a deterioration of the impact strength of the material (Fig.4).

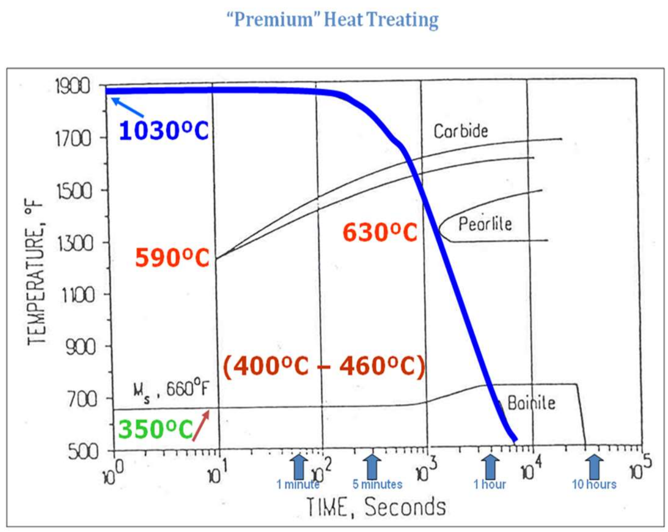



***Fig. 4*** *– CCT diagram H13 steel with critical regions and temperatures*

Therefore, in order for the material to survive this high cooling rate without cracks and extreme deformations, it is necessary that its shape is adapted to this goal. Far more important for the further development of service life, more than deformation, are the structural properties of the material achieved by heat treatment. Carbide precipitation and extreme austenitic grain size growth are one of the main sources of failure. In some cases, extreme grain growth can also cause inhomogeneous deformations of the metallurgical semi-finished product, so it may not always be related to excessive or inappropriately selected austenitization temperature. All parts undergoing high-speed cooling must be equipped with minimum radii of R3-5 mm on all critical shapes, the surface roughness after machining must be treated, and no notches must be created on which stress would be concentrated. Thus, it is common for tool manufacturers to prepare their parts so that they can undergo a high-speed cooling process regardless of deformation. Otherwise, any exception to this rule may cause and usually lead to a deterioration in the mechanical properties of the steel.

The cooling rate is usually not prescribed and the choice is therefore up to the heat treatment supplier, except in cases where heat treatment is ordered according to Nadca. Since this parameter is not even generally stated on the material sheets, it is very difficult to talk about the usual parameter. Therefore, in order to clearly distinguish whether this parameter was chosen appropriately or inappropriately, it is necessary to perform a more objective evaluation of heat treatment result  using impact tests.

After hardening, the material must be tempered at least twice. Despite all efforts to complete the martensitic conversion, the material still contains a large amount of residual austenite. This is due to the position of $\mathbf{M_f}$ and the fact that it is a 3D body with a heterogeneous temperature distribution inside. By the time the surface is cooled to 70 ° C, more than 50% of the residual austenite may still be present inside the body. It is therefore a question of when to start the first tempering when the conversion of austenite to martensite has not yet been completed.

The usual method says that as soon as possible after hardening, as soon as the part reaches a temperature of **50-70 °C**. The problem, however, is that it is during post-cooling that the greatest risk of cracks is due to the fact that we are below $\mathbf{M_s}$ and there is a superposition of stresses both from the temperature difference and from the phase transformation. Therefore, aftercooling in a furnace under a gas stream is usually applied until reaching **Tc = 150 °C** (some steel producers recommend aftercooling in a furnace up to **100 °C**), then only in the open air. The time to reach **Ts = 50 °C** is then tied to the weight and shape of the body.

Therefore, the time definition of the time to the first tempering is an abstract concept and cannot be considered a predefined parameter. However, post-cooling after hardening must be controlled even after removing the part from the furnace. For this reason, a magnetically clamped (usually bimetallic) thermometer is placed on the parts and the surface temperature of the part is measured during the cooling outside the furnace. There is no record of this phase of the process (therefore usually).

After reaching the usual temperature **Ts = 50 °C**, it is possible to start tempering, but this time can take several hours for large tools. Also the view of this temperature **Ts = 50 °C** is different between Nadca and some steel producers. For this reason, the usual procedure will vary according to the client, and may also vary depending on the type of material used.

Tempering must be carried out at least twice, taking into account the not-tempered martensite formed by the decomposition of residual austenite after the first tempering. However, for more demanding applications, it is better to temper all structural components at least twice, and therefore tempering 3 times is required.

## 4. Output control of heat treatment

As important as the incoming inspection of the purchased material condition is to exclude future disputes, so is the inspection of the condition after heat treatment. The inspection is performed again by means of impact tests on a sample of material which, together with the real part, passes through the entire heat treatment process. In the event that the impact tests do not reach the usual KCV values after heat treatment, the heat treater is obliged to carry out corrective heat treatment. There is a general consensus that only one corrective heat treatment is allowed. However, before applying it, the material must be soft-annealed. The annealing conditions follow either the prescription of the steel supplier or Nadca, if specified.

In the event that even repeated hardening does not give adequate results, and at the same time the initial tests of material testing are tolerant, the TZ provider should be obliged to reimburse all costs incurred so far.

| | Annealed | | Hardened and tempered | |
|---|---|---|---|---|
| | Average (J) | Min (J) | Average (J) | Min (J) |
| H13 | 13,6 | 10,8 | 10,8 | 6,1 |
| Thyrotherm 2367 | 13,6 | 10,8 | 10,8 | 6,1 |
| Dievar | 19,0 | 14,9 | 14,9 | 12,2 |
| W403 VMR | 13,6 | 10,8 | 10,8 | 6,1 |
| W300 Isobloc | 19,0 | 14,9 | 14,9 | 12,2 |
| Thyrotherm 2343 Supra | 19,0 | 14,9 | 14,9 | 12,2 |
| Vidar Supreme | 19,0 | 14,9 | 14,9 | 12,2 |
| W400 VMR | 27,0 | 23,0 | 21,7 | 19,0 |
| Vidar Superior | 19,0 | 14,9 | 14,9 | 12,2 |
| TQ-1 | 19,0 | 14,9 | 14,9 | 12,2 |
| Thyrotherm E-38K | 19,0 | 14,9 | 14,9 | 12,2 |
| Aubert Duval ADC3 | 19,0 | 14,9 | 14,9 | 12,2 |

***Tab.3*** *- Theoretical values of toughness for steel documented by Nadca [1]*

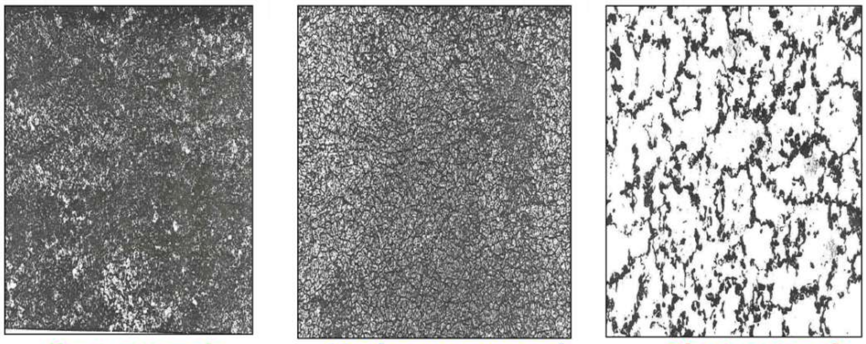



***Fig. 5*** *- Toughness of H13 steel after different cooling speed on samples with the same hardness 45 HRc [3]*

Evaluation of the heat treatment result by measuring the hardness is completely insufficient. It is known that all types of structures after hardening can have the same hardness under certain circumstances, but only some the right toughness, or completely different mechanical properties, both at ambient and elevated temperatures.

If after the heat treatment the hardness is higher than specified, then it is appropriate to include the 4th or other tempering to the required hardness. It is assumed that the contracting authority has exactly documented reasons why it determined such hardness and the supplier of heat treatment is not obliged to examine these reasons.

However, if the hardness after heat treatment is lower than ordered, then it is not possible to make an optimal decision without knowledge of impact strength. Because lower hardness means higher toughness, it is usually more reliable to release the material into use than to rework it repeatedly. However, the criterion that this process can decide is only in impact tests. Therefore, if the customer does not perform these impact tests as the usual method of testing, then it cannot be expected that it could be successful in disputes over heat treatment result complaints.

## 5. Sub-zero treatment

Cryogenic processing of hot work steels is still mainly used for forging tools. The freezing process is usually included immediately after hardening so that a drop in temperature below $\mathbf{M_f}$ allows the phase transformation to be completed. With a further drop in temperature, not only the decomposition of residual austenite is completed, but also the precipitation of secondary carbides, which improve the wear resistance.

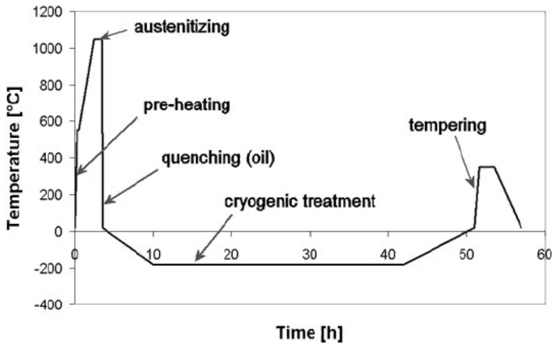



***Fig. 5*** - *Typical cryogenic treatment graph [4]*

In terms of changes in impact strength, no significant changes in its values were found, but a significant improvement in the abrasive properties of the material was found. Therefore, this method of aftercooling is more common with forging tools than with die casting dies. Standard Nadca 207, rev. 2008 does not yet know this process.

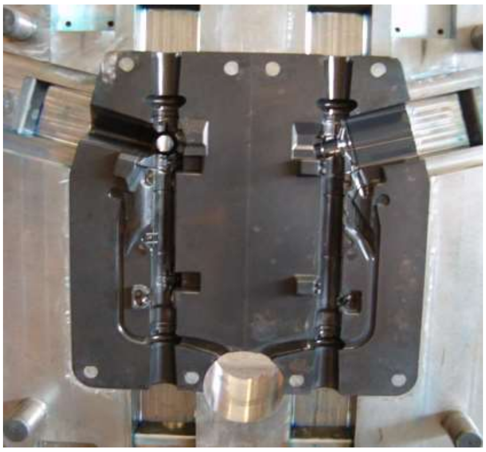
## 6. Surface modification

The optimal processing of steels for hot work has the fundamental task, so that it does not worsen the initial properties of the material. But in no way can he improve them. If the application requires other, specific properties, then they can be solved by other subsequent surface treatments.

These are either nitriding, PVD layers or a combination of both, so-called duplex coatings.

These coatings provide improvements when it is necessary to change the amplitude of tensile to compressive stresses or when it is necessary to prevent increased wear or diffusion of the formed or cast material into the steel. Surface treatments are also important when it is necessary to prevent the material being processed from interacting with the tool material. Die-casting dies are primarily subject of soldering, ie the interaction of the tool surface and Al.

However, processes that mean better running-in properties of the die material, such as controlled oxidation in the case of die casting dies, are also developing successfully. Its advantage is to improve the uniformity of lubricant distribution during tool run-in and thus better cooling of the surface with lower abrasive effect and especially to avoid direct contact of aluminum and steel surface at the beginning of operation, when the die is not yet caught and is prone to initiation thermal fatigue.



*Pic. 6 - Die casting die after process of oxidation*

## 7. Conclusion

Heat treatment is qualified as a special process whose parameters have many variables. These parameters not only have a technical aspect related to the feasibility of the process, they are also related to the usual parameters that affect the legal side of things. Despite the significant improvement in awareness of this problem, it can be stated that constant economic pressure leads many companies to omit basic, albeit usual, operations, and at that moment the legal side of things becomes difficult to defend.

**Literature**

[1] Nadca Publication 229 – Special Quality Die, Steel and Heat Treatment, Acceptance Criteria for Die Casting Dies

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[3] Corwyn Berger - Extended tool life on die casting, Presentation Brno, 1999

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