EFFECT OF AUSTENITISING TEMPERATURE AND COOLING RATE ON MICROSTRUCTURE IN A HOT- WORK TOOL STEEL

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Abstract: The effects on microstructucture of austenitising temperature and cooling rate during hardening were studied for a hot-work tool steel. Transformation temperatures were determined by dilatometry, scanning electron microscopy was used to characterise the microstructure and both retained austenite contents and their lattice parameters were measured by neutron diffraction. For lower cooling rates, lower austenitising temperatures produce larger amounts of both retained austenite and bainite. Retained austenite in bainitic structures is higher in carbon than in martensitic structures. Consequently, lowering the austenitising temperature will affect microstructure and properties.

Keywords: austenitising temperature, bainitic reaction, retained austenite, lattice parameter

1. INTRODUCTION

The development of the automotive industry demands parts of increasingly complex design and dimensions. This implies also growing complexity and dimensions of the tools to produce them. At the same time there is a strive for higher productivity, faster processes, shorter lead times and less production stops. Therefore high-pressure die casting has become a very common way to produce the above mentioned parts. In this process, good hot properties of the tooling material are key for the tool to deliver a performance that fulfils all the aspects enclosed in the idea of “high productivity”. The alloy design and the production process of the tool steel will project on it a certain potential for developing the desired properties. Sometimes aditional steps are added to the conventional ingot- casting production route, such as electro-slag remelting (ESR) or vacuum-arc remelting (VAR). These are nothing else than attempts to increase the potential of the steel to develop the optimal microstructure thereby assuring properties by improving the original cast structure (Roberts, *et al.,* 1994). However, it is actually the heat treatment process which is the decisive step in order for the steel to materialise that embedded potential. A proper heat treatment is a *sine qua non* condition for a high quality tool to deliver top performance. For this reason, the automotive industry is putting a lot of focus on heat treatment processes and their quality, generating in many occasions very demanding specifications on both the tooling material and the hardening process that the tool will undergo before being put into production.

The above mentioned tools present also some particular problems and limitations on the hardening process due to their large dimensions and complex geometry: their surface will be exposed to the hardening temperature for much longer time than the core, and the cooling rate at the core is far from being optimal. This last aspect is due to the slow heat transfer and the need to minimise thermal stresses in order to avoid cracking of the tool during the hardening process.

Tool steel grades for hot-work applications are usually relatively low alloyed and therefore they have a relatively low amount of equilibrium carbides at austenitising temperatures. This often results in grain growth (Porter and Easterling, 2004) during austenitising which will be detrimental to the mechanical properties (Roberts, *et al.*, 1994). This effect is especially pronounced at the surface due to the prolonged soaking time alloys (Chandler, 2000) and it has therefore become practice to lower the austenitising temperature in the case of large tools. The austenitising temperature not only has an effect on the type of tempering carbides that precipitate, which will strongly influence the hot properties (Coll Ferrari, *et al.*, 2013), but it will also have an impact on the bainitic transformation that is to take place in the areas where the cooling rate is low. The aim of the present paper is therefore to study how austenitising temperature affects the microstructure of a hot- work tool steel for different cooling rates.

2. EXPERIMENTAL

A series of heat treatments were carried out in order to analyse the effects of austenitising temperature and cooling rate. Thermodynamical simulations were run to get information about the composition of the austenite formed at the different temperatures. Phase transformations were studied by dilatometry and resulting microstructures were investigated by scanning electron microscopy (SEM). Retained austenite contents as well as their lattice parameters were measured by neutron diffraction.

2.1. Test material

Steel grades for hot-work applications are specially designed for having a high heat conductivity, good heat checking and tempering resistance and optimal mechanical proporties at elevated temperatures. This is usually achieved by having a low alloying content but including some carbide-forming elements, like molybdenum, chromium and vanadium. The material investigated in this paper, Uddeholm Dievar, is a modified version of the standard grade H13 with ESR quality. The nominal chemical composition is presented in Table 1. The steel is suitable for high demand hot- work applications like die casting, forging and extrusion.

Table 1. Nominal chemical composition of Uddeholm Dievar.

Alloying element	C	Si	Mn	Cr	Mo	V
Concentration (wt%)	0.35	0.2	0.5	5.0	2.3	0.6

Samples were hardened and tempered in a Schemtz vacuum furnace with internal dimensions 300×300×600 mm and nitrogen quenching with a maximum overpressure of 5 bars and unidirectional cooling from the top. The samples were heat treated as shown in Table 2. Heat treatment parameters were chosen in order to produce one martensitic and one bainitic microstructure for each of the two austenitising temperatures.

Table 2. Heat treatments.

Sample denomination	Austenitising temperature (°C)	Holding time (min)	Cooling time [800- 500°C] (s)	Condition
M_{980}	980	30	63	Untempered
M_{1025}	1025	30	63	Untempered
B_{980}	980	30	1800	Untempered
B_{1025}	1025	30	1800	Untempered

2.2. Experimental methods

Samples for Scanning Electron Microscopy (SEM) were taken out from both the surface and the center of the original material in the longitudinal direction and heat treated in pairs. No significant differences were found between the surface and the centre, as expected from an ESR- grade, so only results from the centre are reported. The SEM used for the microstructure investigations was a FEI Quanta 600F with a field emission gun. Everhart-Thornley detector (ETD) was used for samples M_{980}, M_{1025} and B_{980}, while a Back Scattered Electron Detector (EBSD) was used for sample B_{1025}. This difference was due to practical reasons.

The dilatometry samples were cylindrical with 4 mm in diameter and 10 mm in length and they were taken out from the center of the original material in the longitudinal direction. The dilatometer was a Bähr DIL805A with Argon cooling.

Samples were also analysed by the means of neutron diffraction in order to obtain the retained austenite content averaged over the sample volume and its lattice parameter. The measurements were carried out at room temperature using the MEREDIT@NPI instrument. Each measurement had the same time delay and settings. Neutron diffraction patterns were collected between 4 and 144° of 2θ with steps of 0.08°. A neutron wavelength of 1.4618 Å was selected from the primary beam by a mosaic copper monochromator. Data refinements and structure phase analysis were performed with full pattern fitting method using the FullProf software.

3. RESULTS

3.1. Dilatometry

Two samples were run in the dilatometer, reproducing the heat treatments performed on samples B_{980} and $B_{1025.}$ The changes in the samples' length (ΔL) with temperature were recorded in order to collect information about the phase transformations that take place, and their start and stop temperatures. The resulting curves are displayed in Figure 1.

Both curves start with the initial value of ΔL=0 at room temperature. Heating up until the formation of austenite starts yields a growing ΔL which is a result of thermal expansion of the material. The following shrinkage is generated by the transformation of ferrite into austenite (Roberts, *et al.,* 1994) which has a more close packed structure (Apraiz Barreiro, 2002). When the austenitic transformation is completed the material will experience a new thermal expansion due to temperature increase until reaching the chosen austenitising temperature (Roberts, *et al.,* 1994). Quenching after holding time causes thermal contraction until the bainitic transformation starts. This transformation produces an expansion in the sample (ΔL_B). The bainitic transformations start (B_s) and finish (B_f) temperatures are higher for the higher austenitising temperature and the expansion is larger for sample B_{980}. This suggests that the amount of bainite formed in sample B_{980} is greater than that in sample B_{1025}. After the bainitic transformation is concluded, a new transformation also producing an expansion(ΔL_M) of the sample, takes place. This is a martensitic transformation and is most clearly seen for sample B_{1025}. The temperature at which it starts is here denominated M_s and the temperature at which it finishes is refered to as M_f. Sample B_{980} undergoes a similar transformation, but of such a small extension that is negligible.

Both samples had shrunk at the end of the quenching, compared to their initial dimension. This points to the presence of a phase with higher density. This phase is probably retained austenite, and the greater total shrinkage (ΔL_{TOTAL}) in sample B_{980} suggests a greater content of this phase in the mentioned sample.

The starting and finishing temperatures for all phase transformations produced during quenching and corresponding dimensional changes are displayed in Table 3.

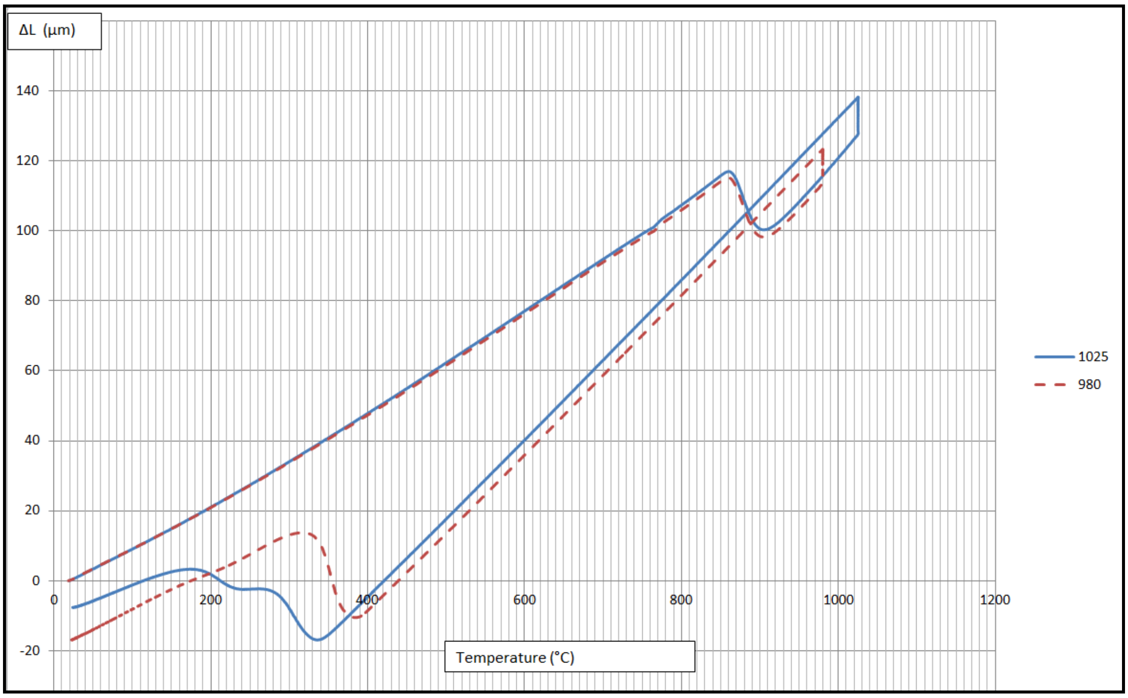

Fig. 1. Dilatometry curves. Variation of ΔL with temperature for similar heat treatments as for samples B_{980} and B_{1025}.

Table 3. Transformation temperatures and length variations obtained by dilatometry.

Austenitising temperature (°C)	980	1025
B_s (°C)	387	335
B_f (°C)	309	267
ΔL_B (µm)	+24.02	+14.53
M_s (°C)	230	237
M_f (°C)	177	156
ΔL_M(µm)	~0	5.25
ΔL_{TOTAL}(µm)	-17.13	-7.65

3.2. Retained austenite

Retained austenite contents and lattice parameters in the as-quenched condition were measured with neutron diffraction. The results are shown in Table 4.

Bainitic microstructures contained a higher amount of retained austenite than the martensitic ones. The lower austenitising temperature resulted in larger amounts of retained austenite, especially for the bainitic samples. At the same time, the lattice parameter of the retained austenite is greater in the case of a bainitic structure compared to those obtained in martensitic structures. This points to a higher alloying content, most likely higher C-content, of the austenite in the bainitic structure.

Table 4. Retained austenite content, its lattice parameter and hardness.

Sample	Retained austenite content (%)	Lattice parameter of retained austenite (Å)
M_{980}	5.7±0.2	3.6052 ±0.0014
M_{1025}	4.6±0.2	3.6006±0.0016
B_{980}	18.1±0.2	3.61414±0.004
B_{1025}	10.5±0.2	3.61419±0.0072

3.3. Microstructures

Austenite is the stable phase of iron at elevated temperatures. Its carbon solubility increases with temperature and is much higher than for ferrite. Therefore, the carbon dissolved into the austenite during austenitisation in a hardening process, increases with increasing hardening temperature. For the same reason, the amount of undissolved carbides to be found in the microstructure decreases with higher austenitising temperatures (Pero-Sanz Elorz, 1995).

When the quenching is fast enough the austenite will transform into martensite, which is a metastable phase. On the other hand, when quenching is performed so that the combination of cooling and diffusion kinetics are such that a certain rearrengement of the carbon takes place, bainite which is composed of ferrite (low in carbon) and cementite (rich in carbon) form (Bhadeshia and Honeycombe, 2006).

SEM investigations showed that the austenitising temperature has an effect on the distribution and morphology of the phases in both martensitic and bainitic microstructures.

In the martensitic structures (samples M_{980} and M_{1025}), the more heavily etched areas, probably martensite, are smaller when austenitising at 980°C compared to 1025°C . There are also many more undissolved carbides for the lower austenitising temperature, which appear as small dots in the micrograph, mostly of light gray colour or white. At the same time, the needle-shaped areas tend to be narrower and longer in the case of the higher austenitising temperature. This can be seen in Figure 2.

In the case of bainitic structures (samples B_{980} and B_{1025}), a higher austenitising temperature yielded less but substantially longer bainitic sheaves than those obtained when hardening using a lower austenitising temperature. This difference can clearly be seen in Figure 3.

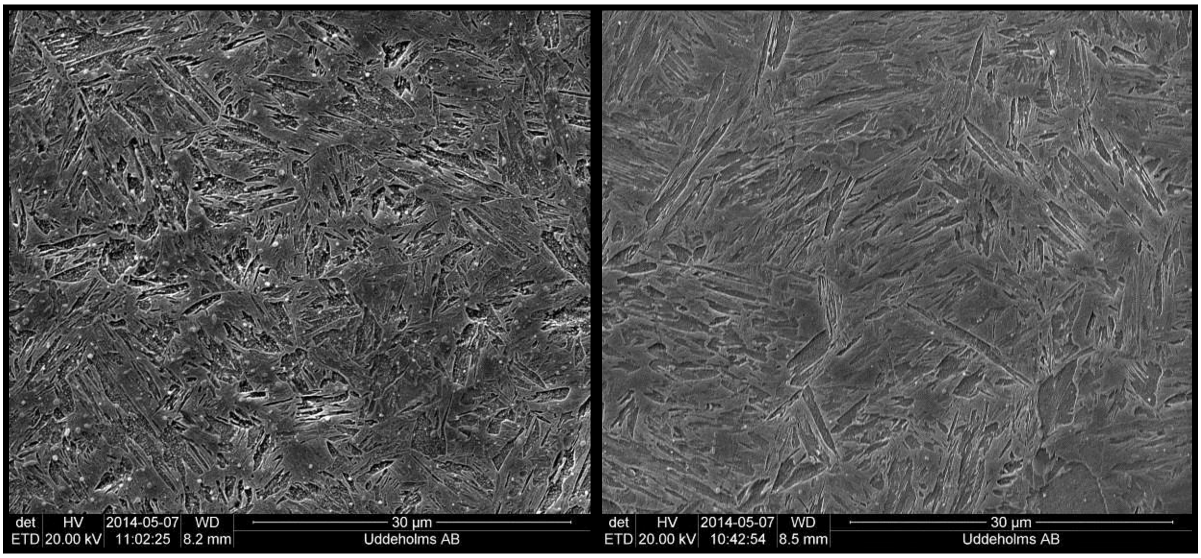

Fig. 2. SEM images of samples M_{980}(to the left) and M_{1025} (to the right).

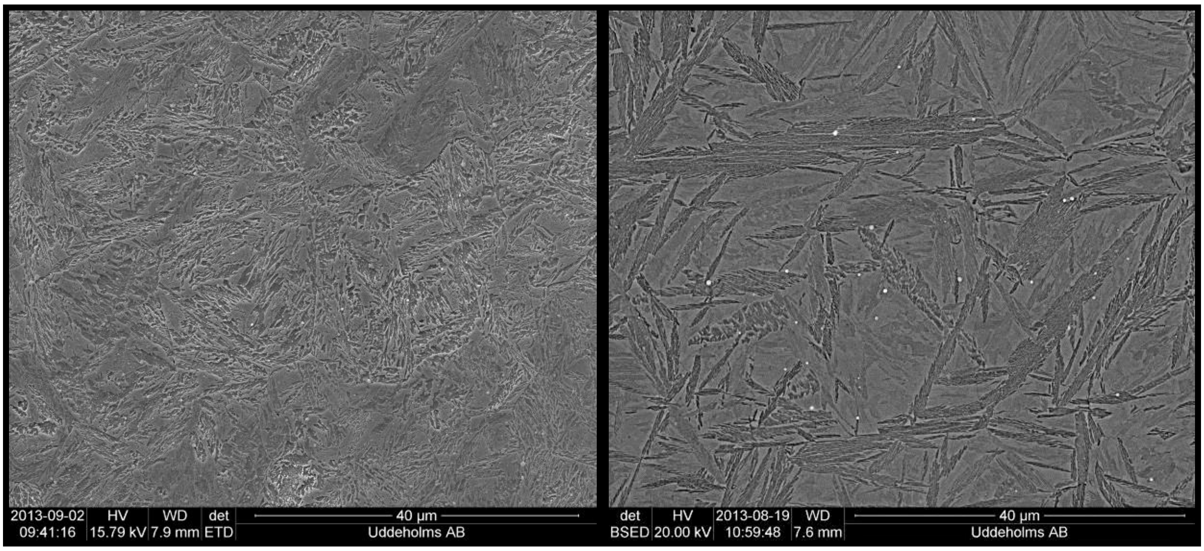

Fig. 3. SEM images of sample B_{980} (to the left) and B_{1025}(to the right).

3.4. Thermodynamic simulations

The calculated compositions of the equilibrium austenite at the two austenitising temperatures are given in Table 5. As can be seen a higher austenitising temperature yields a more highly alloyed austenitewith higher . C, Mo and V contents.

Table 5. Calculated equilibrium austenite composition (wt%) for the different austenitising temperatures.

Temperature (°C)	C	Mo	V	Si	Cr	Mn
980	0.33	2.2	0.4	0.2	5.0	0.5
1025	0.35	2.3	0.5	0.2	5.0	0.5

4. DISCUSSION

The thermodynamic simulations predicted an increasing alloying content of the equilibrium austenite with increasing austenitising temperatures. This would imply a lower amount of undissolved carbides for the higher austenitising temperature (Coll Ferrari, *et al.*, 2013), which agrees with the SEM investigations. Also the lattice parameters of the austenite formed at higher temperature would be expected to be greater as a consequence of the higher alloying content. This was indeed observed after slow cooling but not after rapid cooling according to the neutron diffraction results shown in Table 4. In the case of the martensitic samples, a higher austenitising temperature yielded slightly lower amount of retained austenite which was also rather unexpected. Further studies are needed to explain these results.

Austenitising temperature had an impact on the microstructure, affecting the distribution, quantity and size of the different phases, both in martensitic and bainitic structures. Bainitic sheaves were much more numerous and of smaller size when austenitising at lower temperatures. According to dilatometry results, temperatures B_s and B_f were higher for sample B_{980} than for sample B_{1025}. This probably implies a greater driving force for formation of bainite during cooling (Hillert, *et al., 2005*). This idea also agrees with the fact that a lower austenitising temperature yields a greater amount of bainite (also proven by dilatometry). Dilatometry results also proved that when cooling rate is such that it will produce a mainly bainitic structure, many transformation characteristics are affected by the austenitising temperature. These include the temperatures at which the bainitic transformation starts and finishes, the amounts of bainite and martensite formed and the retained austenite content.

In the case of mainly bainitic samples, a lower austenitising temperature yielded a much higher retained austenite content. This can be explained by the fact that during bainitic transformation carbon will redistribute and enrich the adjacent austenite and thereby make it more stable. The carbon content in the retained austenite in these bainitic structures is therefore the result of two different enriching mechanisms: first, the dissolution of carbon into the matrix that takes place during austenitising. This factor increases with growing austenitising temperatures (see Table 5). The other enriching mechanism is, as explained, the diffusion of carbon during the bainitic transformation. By combining the data in Table 4 and Table 5 it can be concluded that for a heat treatment corresponding to sample B_{1025} the carbon enrichment of retained austenite during bainitic transformation is greater than for theheat treatment corresponding to sample B_{980}.

Compromising on the austenitising temperature in order to avoid grain growth in large tools, as discussed in the introduction, not only affects the hot properties of the material, but also its microstructure. For this reason, the impact of these effects on the mechanical properties needs to be studied further.

5. CONCLUSIONS

The austenitising temperature as well as the cooling rate during hardening influence the fractions and distribution of phases in the microstructure. Increasing austenitising temperature increases the alloying content in the austenite and decreases the amount of undissolved carbides.

Lowering the austenitising temperature from 1025°C to 980°C increases B_s- and B_f- temperatures and also the amount of bainite.

Lowering the austenitising temperature yields a greater amount of retained austenite, especially in the bainitic structures.

The lattice parameters of the retained austenite in the bainitic structures were larger than in martensitic structures. This suggests that the retained austenite in bainite had a higher carbon content.

The carbon enrichment of retained austenite during bainitic transformation is greater in sample B_{1025} than in sample B_{980}.

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