EFFECT OF AUSTENITISING TEMPERATURE ON AUSTEMPERING KINETICS OF Ni-Mo ALLOYED DUCTILE IRON

VPLIV TEMPERATURE AVSTENITIZACIJE NA KINETIKO AUSTEMPERING LEGIRANE NODULARNE LITINE

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The effect of austenitising temperature on the transformation kinetics of Fe-3.2C-2.68Si-0.07Mn-0.022P-0.003S-0.02Cu-0.12Cr-0.8Ni-0.25Mo [mass fractions] ductile iron was investigated. X-ray diffraction and optical microscopy were used to determine the transformation kinetics during the austempering at 350 °C after austenitising at 850 °C, 900 °C and 930 °C for 120 min. The stage I reaction of austempering occurs in two steps, first in eutectic cells and than in the intercellular area. Decreasing the austenitising temperature is shown to increase the driving force for the stage I reaction but to have little effect on the stage II reaction. By lower austenitising temperature the rate of transformation is greater, a more uniform microstructure is obtained, while the austemperability is smaller. These changes shift the processing window for Ni-Mo ductile iron for selected austenitising temperature.

Key words: ductile iron, austenitisation temperature, austempering kinetics, microstructure

Raziskan je bil vpliv temperature avstenitizacije na kinetiko premene nodularne litine Fe-3,2C-2,68Si-0,07Mn-0,022P-0,003S-0,02Cu-0,12Cr-0,8Ni-0,25Mo. Kinetika premene pri 350 °C po avstenitizaciji pri 850 °C, 900 °C in 930 °C je bila določena z optično mikroskopijo in difrakcijo rentgenskih žarkov. Prva stopnja premene se izvrši v dveh fazah, najprej v evtektičnih celicah in nato med temi celicami. Pri znižanju temperature avstenitizacije se poveča gonilna sila za prvo stopnjo premene, ki ima majhen vpliv na drugo stopnjo premene. Pri nižji temperaturi avstenitizacije je večja hitrost premene in nastane bolj enakomerna mikrostruktura, vendar je manjša sposobnost litine za avstempering. Te spremembe premaknejo procesno okno za nodularno litino Ni-Mo za izbrano temperaturo avstenitizacije.

Ključne besede: nodularna litina, temperatura avstenitizacije, kinetika premene, mikrostruktura

1 INTRODUCTION

The heat-treating of the ductile cast iron produces the austempered ductile iron (ADI) with outstanding mechanical properties that can be varied over a wide range by changing the heat treatment parameters. ADI have an excellent combination of strength, fracture toughness and wear resistance and are used for a wide variety of applications in automotive, rail and heavy engineering industries [1-5]. Presently ADI have only a small fraction of the ductile iron market. The market is expected to grow with machining and heat-treatment facilities available in more foundries.

The heat treatment of austempering consists of austenitising ductile iron in the temperature range of 850–950 °C, quenching to the austempering temperature in the range of 250–400 °C for a controlled time and cooling to room temperature. At austenitising the as-cast matrix structure is transformed either into austenite or a mixture of proeutectoid ferrite and austenite. The austenitising step in the treatment of ductile iron differs from that of steel because the austenite carbon content in an iron (0.6–1.1 %) depends on the iron composition and austenitising temperature, while it is not case for the austenite carbon content in steel because it doesn't depend on austenitising temperature.

During the austempering process, ADI undergoes a two-stage transformation [4-5]. In the first stage, the austenite (γ) is transformed into bainitic ferrite (α) and carbon enriched austenite (γ_{hc}), a product known as ausferrite with the reaction:

$$\gamma \rightarrow \alpha + \gamma_{hc} \qquad (1)$$

If the casting is held at the austempering temperature for too long, then the carbon-enriched austenite (γ_{hc}) further decomposes into ferrite (α) and carbide, thus:

$$\gamma_{hc} \rightarrow \alpha + carbide \qquad (2)$$

Transitional carbide, such as ε-carbide, is often form in the early stages of austempering and it transforms to the non-coherent carbide Fe_3C by longer isothermal quenching.

The optimal mechanical properties are achieved upon completion of the first reaction, but before the second reaction is started, since, this reaction produces carbide precipitates, which make the material very brittle. This reaction is, therefore, undesirable and must be avoided.

The time period between the end of stage I and the onset of stage II is called as processing window.

In ductile iron, tight control of austenitising temperature is imperative if consistent properties are to be obtained. Variations in austenitising temperature shift the processing window over the threshold of optimal properties. In general, the increasing of austenitising temperature delays the austempering transformation and shrinks the size of processing window. Thus, the austenitising temperature has a significant effect on the austemperability and transformation behavior at higher temperatures, when the matrix carbon content increases and the austenite grain size is increased. This increase augments the austemperability and reduces the rate of reaction allowing in this way the treatment of larger castings and the use of less efficient quenching baths [5].

Lower austenitising temperatures may be specified for castings, which are to be machined after austempering. With lowering the austenitising temperature the volume of untransformed austenite is diminished. Under the higher pressures involved in machining, the untransformed austenite could transform to martensite and in this case the machinability is decreased. On the other hand, a decrease in austenitising temperature frequently results in incomplete austenitisation, which leads to lower ADI properties.

The main objective of the present work was to study the effect of austenitising temperature on the austemperability of Ni-Mo alloyed ductile iron with examination of microstructure, transformation kinetics and to determine the processing window.

2 EXPERIMENTAL

2.1 Preparation and examination of specimens

The ductile iron used in the present work was produced in a medium frequency induction furnace of 100 kg capacity. The charge was consisted of 50 % pig iron ingot and 50 % low manganese steel scrap. The melt was treated with 1.8 % of magnesium ferrosilicon alloy using the sandwich technique. The melt was then inoculated with 0.67 % foundry grade Fe-Si with nominally 75 % Si and cast at 1400 °C into the a standard Y-block mould, which ensured a sound casting.

Image analysis methods were used to measure the volume fraction of ferrite, pearlite, carbides and graphite and for the nodule counting in the as-cast structures.

2.2 Austemperability

The effect of alloying additions and austenitising temperature on austemperability (critical bar diameter that can be austempered without pearlite formation) was also investigated in this work. Voight and Loper [5] suggested the following regression function for the calculation of the critical bar diameter

$$D_c = 124 C_\gamma^\circ + 27(\% \text{ Si}) + 22(\% \text{ Mn}) + 16(\% \text{ Ni}) +$$
$$+ 25(\% \text{ Mo}) + 1.68 \times 10^{-4} T_a^2 + 12(\% \text{ Cu})(\% \text{ Ni}) +$$
$$+ 62(\% \text{ Cu})(\% \text{ Mn}) + 88(\% \text{ Ni})(\% \text{ Mo}) +$$
$$+ 11(\% \text{ Mn})(\% \text{ Cu}) + 127(\% \text{ Mn})(\% \text{ Mo}) -$$
$$- 20(\% \text{ Mn})(\% \text{ Ni}) - 137 \qquad (3)$$

where C_γ° is the C content in matrix austenite at the austenitising temperature and T_a is the austempering temperature. The content of carbon in austenite depends on the austenitising temperature T_γ and the iron composition. This dependence is frequently described by the approximate expression [5]

$$C_\gamma^\circ = T_\gamma / 420 - 0.17(\% \text{ Si}) - 0.95 \qquad (4)$$

2.3 Heat treatment

Test specimens were initially austenitised for 2 h at 850 °C, 900 °C and 930 °C in a muffle furnace, austempered in molten salt bath at 350 °C for (1, 10, 30, 60, 120, 240, 360, 480) min and finally air-cooled to room temperature.

After austempering the possible decarburised skin was removed with surface grinding. In order to avoid the transformation of metastable austenite to martensite the grinding speed was kept as low as practical.

2.4 Kinetics of Austempering

The specimens for the determination of the kinetics were machined from the test section of the Y-block. The cutting was performed slowly and under liberal cooling to minimize heating and machining stresses. The specimens were than ground to 1200 grade using water lubricated silica carbide paper and etched in a solution of 90 % hydrogen peroxide and 10 % concentrated sulfuric acid for 10–15 min. This treatment removed the work-affected surface and minimized the occurrence of textured structure.

The volume fraction of retained austenite was determined with X-ray diffraction analysis using a Cu-Kα radiation at 40 kV and 20 mA and X-rays intensity strip chart recording in the 2θ range from 20–120°. The recorded profiles were computer analyzed to obtain the precise peak positions and the integrated intensities. The volume fraction of retained austenite X_γ was determined by the direct comparison method using integrated intensities of the (110) and (211) peaks of ferrite, and the (111), (220) and (311) peaks of austenite. The carbon content in austenite was deduced using the relationship [6].

$$a_\gamma = 0.3548 + 0.0044 \, C_\gamma \qquad (5)$$

where a_γ/nm is the austenite lattice parameter and C_γ/% is mass fraction of its carbon content. The austenite (111), (220) and (311) peaks were used to deduce the lattice parameter.

Specimens for light microscopy were prepared by the standard metallographic technique. In austempered duc-

tile irons it is difficult, even for an expert eye, to identify the various phases on black and white micrographs. For a more reliable phase identification also heat-tinting technique was used. The tinting of samples after light etching in 2 % nital improved the phase resolution and was achieved by heating the specimens in air at 260 °C for 5.5 h and cooling to room temperature. With this technique the various phases appear in colors of different intensity. These colors are: untransformed austenite - light blue, reacted high carbon austenite – light brown, ferrite – beige, eutectic carbide – white or cream, martensite – dark blue. For point counting at last 2000 points were counted at magnification of 500 times. The number of counts was increased at decreased volume fraction of untransformed austenite to maintain a low standard deviation.

3 RESULTS AND DISCUSSION

3.1 Characteristics of as cast irons

The chemical composition of the iron investigated is given in **Table 1** and its microstructural characteristics in **Table 2**. The as cast structure was the typical bullseye type with ferrite surrounding graphite nodules in pearlitic matrix with some carbides at intercellular boundaries. The results in Table 2 are the averages from various locations on the specimens in shape of discs (ϕ = 25 mm).

Table 1: Chemical composition of the investigated ductile iron (mass fractions, %)
Tabela 1: Kemična sestava nodularne litine, uporabljene pri raziskavi

C	Si	Mn	P	S	Cu	Cr	Ni	Mo
3.20	2.67	0.07	0.022	0.003	0.02	0.12	0.80	0.25

Table 2: Microstructural characteristic of the as-cast ductile iron (volume fractions, %)
Tabela 2: Značilnosti mikrostrukture

Graphite	Pearlite	Ferrite	Carbide	Nodule count mm^{-2}
12	37	50	0.08	128

The austemperability is presented in **Figure 1** as the critical bar diameter that can be austempered without pearlite formation. The second dashed curve in **Figure 1** represents previous measurements on unalloyed ductile iron austempered at 350 °C [7]. The critical diameter of ductile iron significantly increases as the content of alloying additions and austenitising temperature increase.

3.2 Influence of austenitising temperature on austempering kinetics

The austenitising temperature is varied with the aim to control the equilibrium of carbon in solution content in austenite, which, in turn influences the austempering kinetics. The retained austenite volume fraction and the
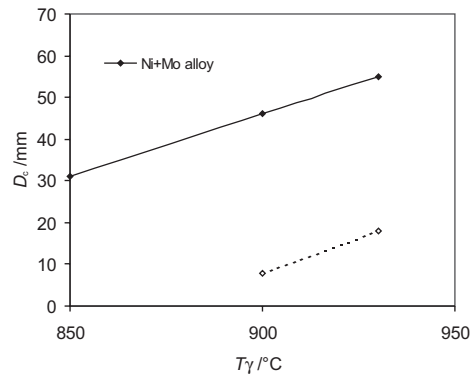

Figure 1: Dependence of the critical bar diameter D_c that can be austempered without pearlite formation on austenitising temperature T_γ for Ni-Mo iron and unalloyed iron austempered at 350 °C (Ref. 7)
Slika 1: Odvisnost kritičnega premena palice D_c, ki se lahko austempra brez nastanka perlita na temperaturo avstenitizacije T_γ za Ni-Mo in nelegirano nodularno litino (Ref. 7)

average austenite carbon content are both determined with X-ray diffraction. The variations of volume of retained austenite (X_γ), austenite carbon content (C_γ), total carbon austenite content ($X_\gamma C_\gamma$), and untransformed austenite, as function of the austempering time for the treated ductile iron are shown in **Figure 2 a-d.** After the high austenitising temperature of 930 °C a relatively higher volume of retained austenite is obtained when compared to austenitising at lower temperature. However, in this case the lowest austenite carbon contents is obtained at all austempering times, while the iron austenitised at 850 °C contains a lower level of retained austenite but the highest carbon contents in retained austenite.

The total austenite carbon contents in the initial austenite matrix increases with the austempering time to a maximal level, which is greater for the higher austenitising temperature, **Figure 2c**. This confirms that less of carbon content is dissolved in austenite at lower austenitising temperature.

The completion of the stage I reaction is very important because it defines the initial time of the processing window and it can be followed by a fast decrease of the content of untransformed austenite content. The residual level of untransformed austenite is related to the delay of the stage I reaction in the intercellular area and associated with the presence of molybdenum. The residual austenite can be transformed to martensite at the cooling to room temperature. This may be the main reason for the difficult achievement of high ductility in molybdenum alloyed irons austempered at high austempering temperature (upper bainite range) [8-9]. The small content of molybdenum (0.25 %) in the investigated ADI could not significantly affect the transformation of austenite at austempering.

The curves that show the untransformed austenite content as a function of austempering time for the investigated ADI are of two different types, one describing
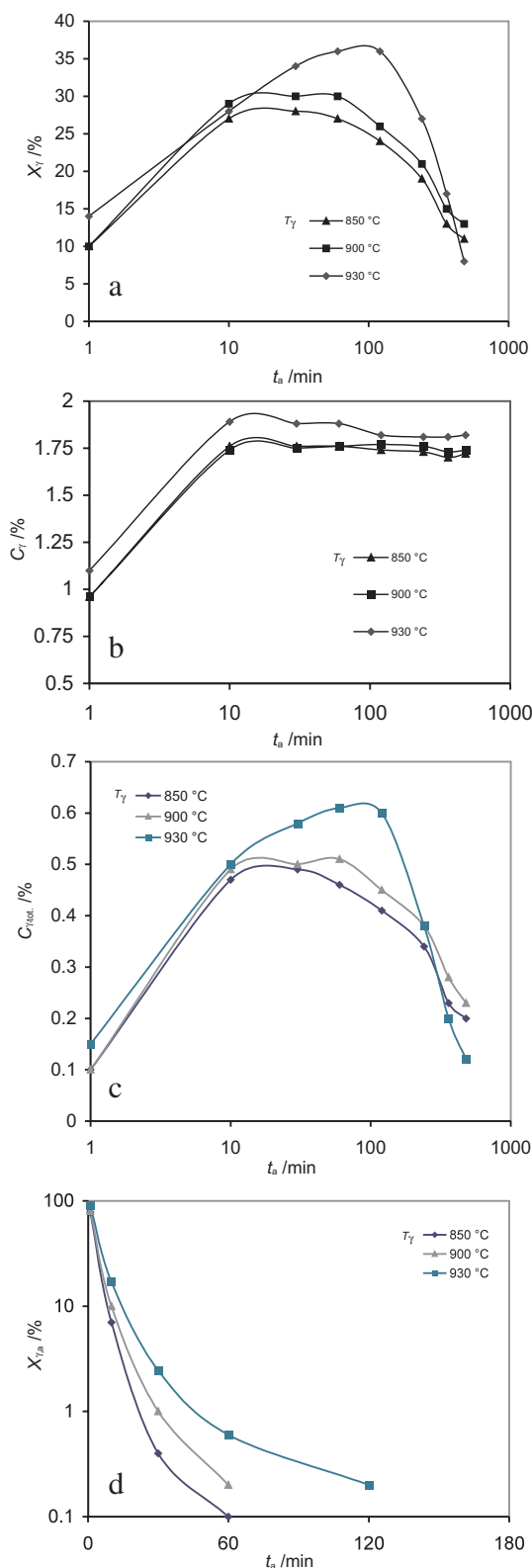
the stage I reaction in the eutectic cell and the second characteristic for the stage I reaction in the intercellular area at longer periods of austempering time. The completion of stage I reaction in eutectic cells can be estimated with extrapolation. This extrapolation shows that the stage I reaction in eutectic cell is completed (the volume of untransformed austenite is 1 %) after 17 min to 55 min. Figure shows also that for lower austenitising temperature 850 °C the time for the completion of the stage I reaction in eutectic cell is decreased.

The decrease in untransformed austenite content for longer periods of austempering time is shown in **Figure 2d.** The time for the completion of the stage I reaction in intercellular region shows a strong dependence from the austenitising temperature. By lower austenitising temperature the level of untransformed austenite content in the intercellular region before completion of the stage I reaction is diminished. In contrast to the stage I kinetics,
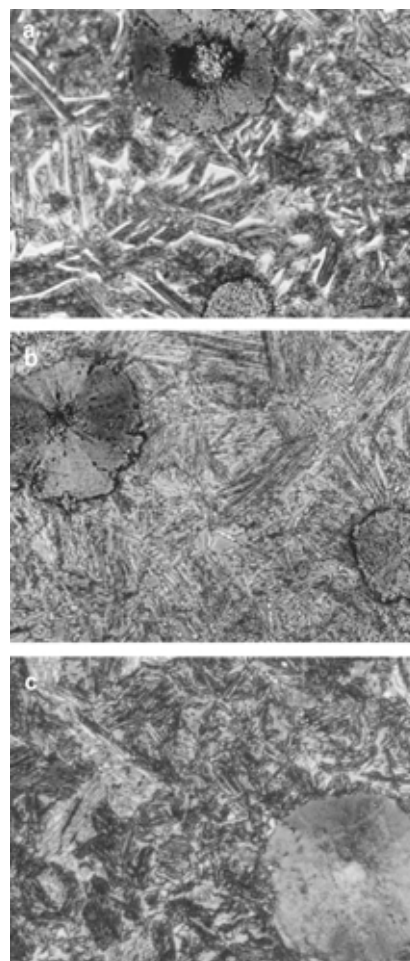

Figure 2: Dependence of: a) volume fraction X_γ of retained austenite; b) austenite carbon content C_γ; c) total carbon austenite content $C_{\gamma,\text{tot.}}$ and d) untransformed austenite $X_{\gamma,\alpha}$ on austempering time t_a and austenitising temperature T_γ
Slika 2: Odvisnost a) volumskega deleža X_γ zaostalega avstenita, b) vsebnosti ogljika C_γ v avstenitu, c) skupne vsebnosti ogljika $C_{\gamma,\text{tot.}}$ in d) netransformiranega avstenita $X_{\gamma,\alpha}$ od časa premene t_a in temperature avstenitizacije T_γ

Figure 3: Effect of austenitising temperature on microstructure of specimens austempered at 350 °C for 60 min, after austenitising at a) 850 °C, b) 900 °C and c) 930 °C for 120 min (Optical micrographs, etching 2 % nital, magnification 300 times)
Slika 3: Vpliv temperature avstenitizacije na mikrostrukturo vzorcev, ki so bili austemprani 60 min pri 350 °C po 120-minutni avstenitizaciji pri a) 850 °C, b) 900 °C in c) 930 °C (optični posnetki, jedkano 2 % nital, pov. 300-kratna)

there does not appear to be any systematic dependence on austenitising temperature for stage II reaction.

3.3 Influence of austenitising temperature on microstructure

The morphology of the matrix after austempering depends on the austenitising temperature and austempering time. In **Figure 3a-c** the microstructure after austenitising at 850 °C, 900 °C and 930 °C for 120 min respectively, and austempering at 350 °C for 60 min is shown. In all cases the microstructure consists of an austenite matrix and upper bainite with is relatively coarse ferrite needles. The microstructure of the specimen austenitised at 850 °C consists of different shares of proeutectoid ferrite, bainitic ferrite and austenite (**Figure 3a**). The presence of proeutectoid ferrite is explained in terms of low austenitising temperature, thus, the austenitising of ADI in the austenite-ferrite-graphite region of the phase diagram in **Figure 4** [10].

Figure 2a shows that for the increase of austenitising temperature from 850 °C to 930 °C the retained austenite content increases from 26 % to 37 %. This increase can be best explained by considering the free energy composition diagram for austenite, ferrite and cementite phases, since, as shown in **Figure 4** if the matrix carbon content decreases then the driving force for the transformation austenite to metastabile ferrite and high carbon austenite increases. The higher driving force allows more nuclei to form and leads to more rapid transformation. Thus, for given austempering tempera-
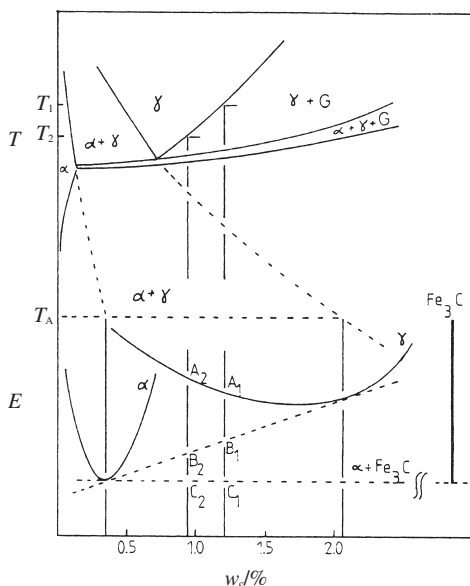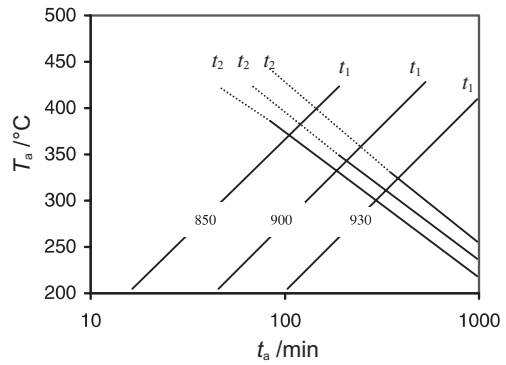

Figure 5: Effect of austenitising temperature T_a on the times t_a of the beginning and the end of the processing window
Slika 5: Vpliv temperature avstenitizacije T_a na čas t_a začetka in konca procesnega okna

ture and time, the bainite transformation reaction is more advanced in a more homogeneous microstructure obtained with austenitising at lower temperature.

3.4 Processing window-microstructural observation

The concept of the processing window was introduced by Rundman [11] to define the austempering time at which optimum mechanical properties, particularly ductility, were obtained. The beginning and the end of the processing window are defined in terms of the amount of untransformed austenite in stage I reaction and the amount of bainitic carbide in stage II reaction.

The criterion used to define the beginning of the window (t_1) is the untransformed austenite volume of 3 % determined from the relationship untransformed austenite volume versus the austempering time using quantitative metallography. The criterion used to define the end of the processing window (t_2) is 90 % of the maximum volume of retained austenite, determined with X-ray diffraction [12]. The horizontal line is drawn at a value of 10 % below the level of the largest content of retained austenite, intersecting the curve at times t_{min} and t_{max}. The value of t_2 is then defined:

$$\ln t_2 = (\ln t_{min} + \ln t_{max})/ 2 \qquad (6)$$

In **Figure 5** the effect of austenitising temperature on the processing window (the difference between the times t_1 and t_2) is shown. For the increased austenitising temperature t_1 is increased, while the change of t_2 is very slight. By lowering the austenitising temperature increases the driving force for the stage I reaction is increased and the processing window is moved to earlier austempering times.

4 CONCLUSIONS

The effect of austenitising temperature on austenite transformation by austempering of alloyed ductile iron was investigated. Specimens of ADI of composition Fe-3.2C-2.68Si-0.07Mn-0.022P-0.003S-0.02Cu-0.12Cr-

Figure 4: Schematic representation of the Fe-C-Si phase diagram and free energy curves for ferrite (α), austenite (γ) and cementite (Fe₃C) phases [10] (the content of carbon $W_c/\%$)
Slika 4: Shematični prikaz faznega diagrama Fe-C-Si in krivulje proste energije E za ferit (α), avstenit (γ) in cementit (Fe₃C) (vsebnost ogljika $W_c/\%$)

0.8Ni-0.25Mn (mass fractions (%) were austenitised at 850 °C, 900 °C and 930 °C and austempered at 350 °C. The transformation kinetics was investigated with X-rays diffraction and optical microscopy. The following conclusions are proposed:

1. With lower austenitising temperature the austenite carbon content and austemperability are diminished and the propensity to the formation proeutectoid ferrite and pearlite by austempering is increased.

2. By decreased the austenitising temperature the driving force for the stage I reaction is increased without systematic effect on the stage II reaction.

3. For lower austenitising temperature the processing window is moved to earlier austempering times.

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