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## INFLUENCE OF AGING TEMPERATURE ON MECHANICAL PROPERTIES OF THE PRECIPITATION HARDENED MARTENSITIC MARVAC 300 STEEL

### WPŁYW TEMPERATURY STARZENIA NA WŁAŚCIWOŚCI STALI MARTENZYTYCZNEJ GATUNKU MARVAC 300 UTWARDZANEJ WYDZIELENIOWO

#### Abstract

This paper presents the results of a comprehensive study of precipitation hardened martensitic steel after quenching and aging at a number of selected temperatures. The results of metallographic and dilatometric studies are presented along with the effect of aging temperature on hardness, strength and resistance to cracking. A significant effect of aging temperature on the properties of the material was found.

*Keywords: maraging steel, tensile strength, toughness, aging, dilatometric curve*

#### Streszczenie

W artykule przedstawiono wyniki badań stali martenzytycznej utwardzanej wydzieleniowo po hartowaniu i starzeniu przy kilku wybranych temperaturach. Zaprezentowano wyniki badań metalograficznych, dylatometrycznych oraz wpływu temperatur starzenia na twardość, wytrzymałość i odporność na pęknięcie. Badania wykazały istotny wpływ temperatur starzenia na właściwości badanego materiału.

*Słowa kluczowe: stal maraging, wytrzymałość na rozciąganie, udarność, starzenie, krzywa dylatometryczna*

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## 1. Introduction

In modern equipment, of increasing importance are steels with a strength between 1000 MPa and 2000 MPa. In ferrous alloys, the mechanical properties of such values are obtained by bainitic microstructure [1, 2], but more often by martensitic. Martensite steels can be divided into two groups.

The first group includes those steels hardened mainly with carbon and through the application of heat treatment and plastic working. Increasing carbon content in steel improves the strength of the material, but also decreases its plastic properties and promotes the appearance of brittle fracture – a phenomenon particularly dangerous in structural steels.

The second group is composed of low-carbon maraging steels, which offer a satisfactory combination of mechanical and plastic properties. These are the steels in which the primary strengthening mechanism during heat treatment is the precipitation of intermetallic phases in a matrix of nearly carbonless martensite. Reducing carbon content prevents the formation of carbides and promotes the formation of intermetallic phases. The intermetallic phases precipitate along the dislocations formed during martensitic transformation, i.e. along the laths of martensite. The alloying elements in these steels are nickel and cobalt, introduced in amounts that allow this steel to remain in the class of martensitic grades. These elements additionally promote high ductility. The main hardening elements are molybdenum and titanium, and indirectly cobalt, since cobalt reduces the solubility of molybdenum in  $\alpha$  iron, which is beneficial to increasing the amount of dispersion precipitates [3, 4].

Nickel content in an amount of about 18% increases the hardenability and after the austenitising process these steels are through-hardened, even when cooled in air. The hardness of the nickel, low-carbon martensitic structure is low, which makes this structure suitable for plastic working. An increase in the steel strength parameters is achieved during aging. Yet, it should be remembered that an aging process that is too long or carried out at a temperature higher than that recommended will reduce the mechanical properties – an phenomenon known as alloy overageing [6].

## 2. Test material

The test material was Marvac 300 steel plate with a thickness of 6.5 mm. The chemical composition is given in Table 1.

Table 1

Chemical composition of the tested steel, weight [%]

Ni	Co	Mo	C	Mn	Si	Ti	Al	P	S
18,67	9,40	4.58	<b>0.014</b>	0.14	0.03	0.75	0.12	<0.01	<0.01

Based on the analysis of the content of alloying elements it has been concluded that the material corresponds to the N18K9M5T steel grade produced in Poland in the late twentieth

century [5]. Steels of similar chemical composition produced by foreign manufacturers are known under various trade names such as Nimarc300, 18Ni300 grade or X2NiCoMo18-9-5 [10, 11].

### 3. Microstructure of the tested steel

This steel is characterised by high metallurgical quality as indicated by its chemical composition as well as microscopic examinations. The surfaces of specimens in unetched condition show no sign of distorted non-metallic inclusions. Only in a few of many fields of vision is the presence of single precipitates of the characteristic titanium carbonitrides noted. Figure 1 shows the steel microstructure etched and subjected to four different variants of heat-treatment:

The microstructure after quenching corresponds to a low-carbon lath martensite. EDS studies have shown that, despite some structural differences, the material has the same chemical composition. The structure after quenching from 830°C and aging at 480°C has

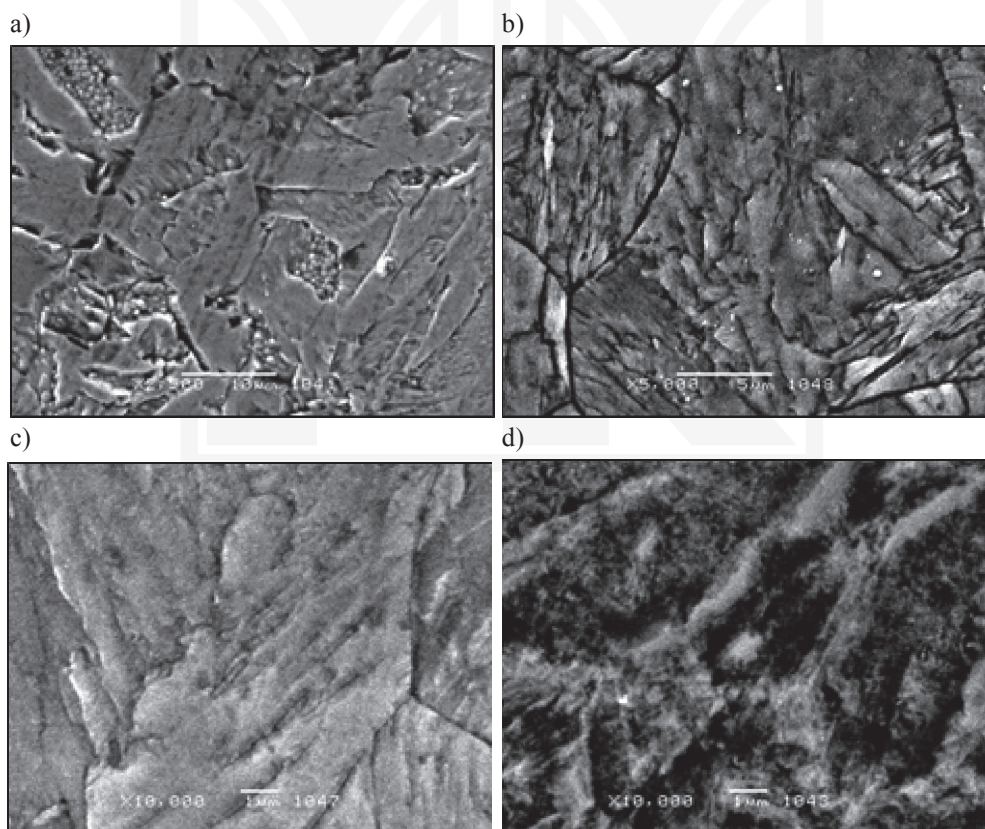


Fig. 1. Steel microstructures: a) quenched from 830°C, b) quenched from 830°C/ and aged at 400°C, c) quenched from 830°C and aged at 480°C, d) quenched from 830°C and aged at 550°C

a more uniform character. One can see the grain boundaries and elements of microstructure that exist inside the grains. The lath character of the martensite is well presented.

Quenching from 830°C with aging at 550°C changes the microstructure of the steel. High magnifications reveal microregions after the presence of very fine precipitates.

#### 4. Dilatometric studies

Samples with a diameter of 4 mm and a length of 35 mm were heated to a temperature of 900°C at a rate of 4°C/min and then cooled at the same speed.

The results of the dilatometric measurements are presented in Fig. 2. Additionally, charts were drawn up to show the dimensional changes and the rate at which those changes occur in time (Fig. 3 – the solid line shows the rate of dimensional changes [%/min] and the dotted line shows the level of dimensional changes occurring in a sample [%]).

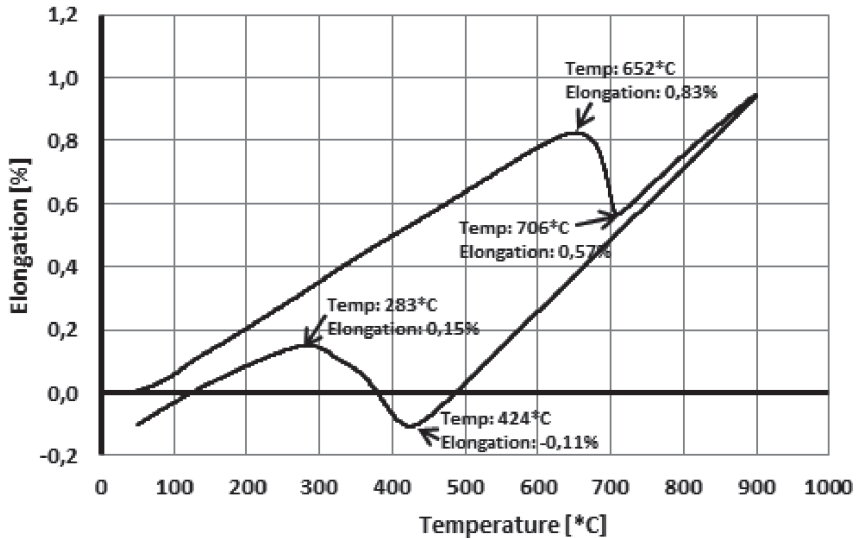


Fig. 2. Dilatogram of sample – the heating and cooling rate was 4°C/minute

In the dilatogram at 652°C one can see the beginning of austenite transformation. The end of the  $\alpha \rightarrow \gamma$  transformation takes place at 706°C. During further heating up to 900°C, no other phase transformations were recorded by the dilatometer. Throughout cooling at a rate of 4°C/minute, the austenite is stable up to a temperature of 424°C.

At this temperature, the undercooled austenite is transformed into a diffusion-free structure of higher specific volume, as evidenced by a clear increase in the sample length. The martensitic transformation ends at the  $M_f$  temperature somewhere about 283°C.

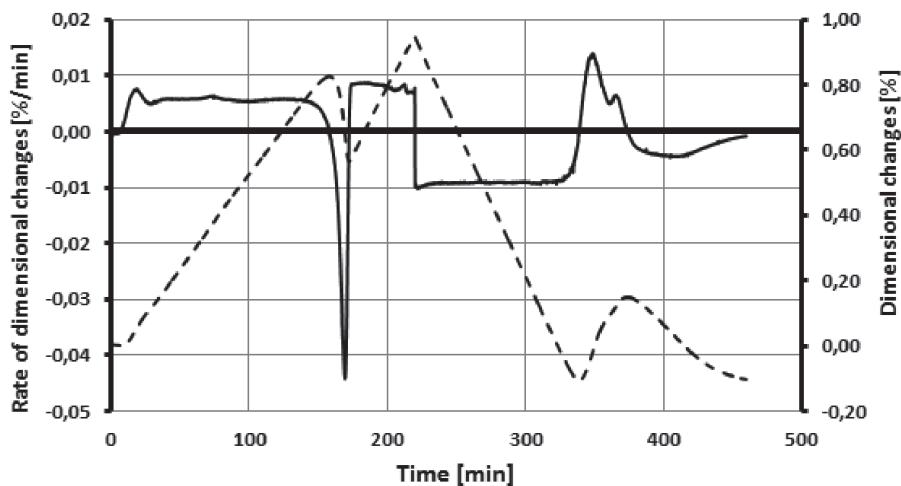


Fig. 3. Dimensional changes and the rate of these changes occurring in a dilatometric sample

## 5. Testing of mechanical properties

The Marvac 300 steel exhibits different properties, depending on the heat treatment applied, and aging temperature in particular [9]. The following shows how six variants of the heat treatment (quenching, and quenching and aging at five different temperatures) affect the hardness, strength and toughness of the steel tested.

The hardness of the samples after quenching was at a level of about 30 HRC. The effect of aging at five different temperatures (300°C, 400°C, 480°C, 550°C and 600°C) on the average steel hardness values is illustrated in Fig. 4. In each case, the aging time was 4 hours.

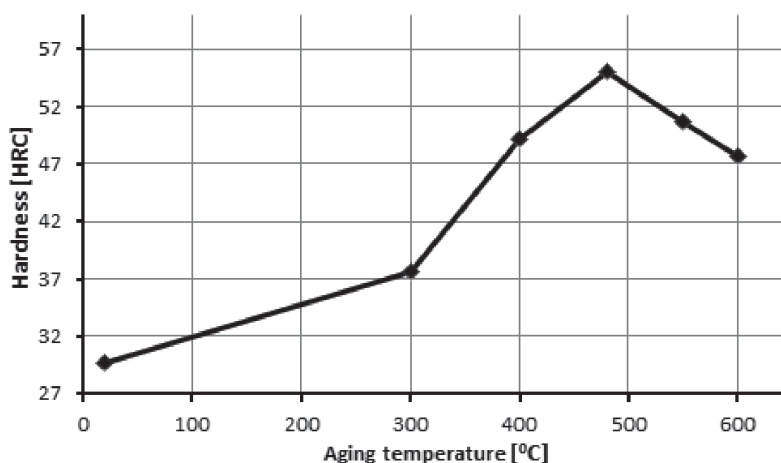


Fig. 4. Aging temperature vs hardness of Marvac 300 steel

The lowest hardness was obtained in the as-quenched state. Aging in the temperature range between 300°C and 480°C made the crystallographic lattice rearrange the atoms of the elements forming intermetallic phases, thus leading to a hardening of the material and hardness increase up to a maximum value of 55HRC at an aging temperature of 480°C. Aging at higher temperatures, i.e. at 550°C and 600°C, reduced hardness by breaking the coherence of the lattice of the precipitating intermetallic phases, at the same time inducing austenite recovery [7, 10].

Aging also affects the strength parameters obtained ( Fig. 5). The steel after quenching is characterised by the lowest values of the yield strength  $R_{0,2} = 915$  MPa and tensile strength  $R_m = 1055$  MPa, combined with high percentage elongation of up to 10%.

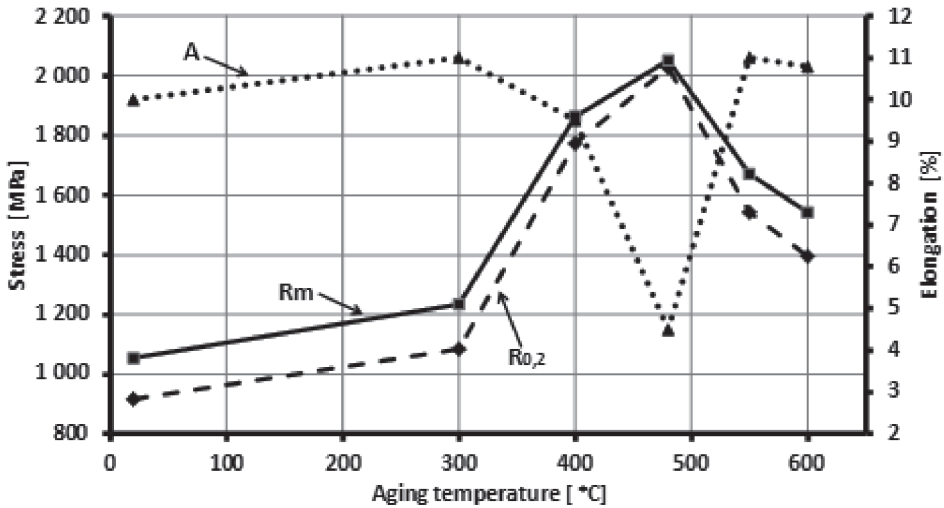


Fig. 5. Yield strength and tensile strength vs aging temperature

The strength starts increasing with the increasing temperature of aging. Its maximum yield strength  $R_{0,2} = 2029$  MPa and tensile strength  $R_m = 2051$  MPa the material reaches at a temperature of 480°C with small percentage elongation of 4.5%.

Aging at higher temperatures of 550°C and 600°C reduces the strength and increases the percentage elongation. At temperatures so high, the aforementioned effect of austenite recovery also takes place, which means that the microstructure now contains, besides the aged martensite, also small but numerous areas of austenite. Austenite on cooling transforms into a low-carbon plastic martensite which results in a loss of strength.

Also deserving of some attention is the change of another parameter referred to in the literature as the “ductility margin”, i.e. the yield strength to tensile strength ratio  $R_{0,2}/R_m$ , which indicates the material’s susceptibility to plastic deformation [8]. Figure 6 shows the lowest value for this ratio after quenching and its maximum after aging at a temperature of 480°C. This confirms earlier observations that Marvac 300 steel has the best plastic properties in as-quenched condition and the lowest susceptibility to plastic deformation after aging at 480°C.

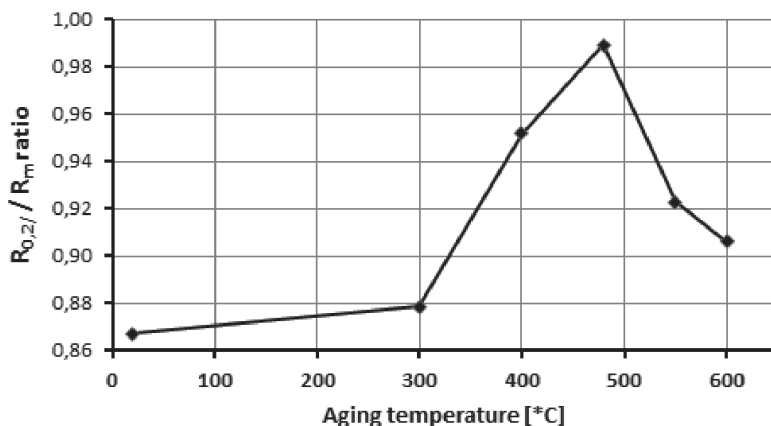


Fig. 6.  $R_{0,2}/R_m$  ratio vs aging temperature

Due to the insufficient thickness of the plate, which prevented the execution of standard impact samples, tests were carried out on samples with dimensions  $5 \times 10 \times 55$  mm, “V” notched to a depth of 2 mm. The samples were broken at ambient temperature.

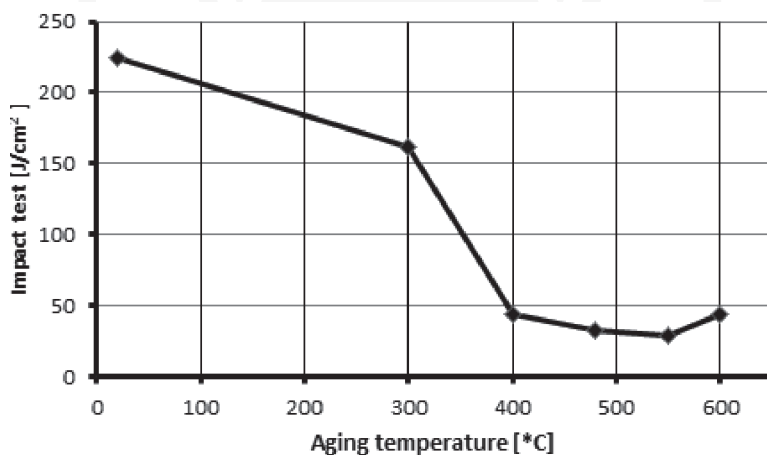


Fig. 7. Toughness vs aging temperature for Marvac300 steel

The plotted graph (Fig. 7) confirms the obvious relationship between the impact test – toughness and temperature of aging. The highest toughness is exhibited by the material in as-quenched condition and the value  $224 J/cm^2$  is calculated per  $1 cm^2$  of the broken surface. Increasing the aging temperature results in a significant decrease toughness. For the temperature of  $400^\circ C$  and higher it is below  $50 J/cm^2$ .



## 6. Discussion of results and conclusions

Microscopic observations and the results of dilatometric studies confirm that even at a very low cooling rate, the steel undergoes a diffusion-free transformation which increases the specific volume of the material. Nickel maraging steel is characterised by small dimensional changes during heat treatment. When heated, these changes are at the beginning of the  $\alpha \rightarrow \gamma$  transformation amount to 0.83%. During cooling, at the start of the  $\alpha \rightarrow \gamma$  transformation, they amount to approximately  $-0.11\%$ .

The highest strength values are obtained after quenching and aging at  $480^\circ\text{C}$ . Aging at  $400^\circ\text{C}$  for 4 hours gives lower values of  $R_m$ , but at the same time it provides a better combination of strength, ductility, and toughness. A distinct reduction in strength and hardness occurs at higher aging temperatures. This phenomenon is due to a coagulation of the precipitated intermetallic phases and breaking of their coherence, all this combined with austenite recovery. Properly controlled austenite recovery may be a beneficial phenomenon. The forming austenite, which at the time of cooling is transformed into plastic martensite, when properly distributed in the microstructure, can improve the ductility of the material.

The largest dynamic loads are transferred by the maraging steel after quenching. In this state, it has also high ductility and good cold workability. Low-carbon nickel martensite, free from precipitates, can offer toughness exceeding  $220 \text{ J/cm}^2$ .

According to the Hollomon-Jaffe parameter, lower annealing temperatures and longer times produce similar effects as annealing at higher temperatures and shorter times. Hence it seems advisable to consider aging at lower temperatures and extended times of heating.

The determination of critical temperatures for  $A_1$  and  $A_3$  and the stability range of the undercooled austenite is useful in designing a thermo-plastic treatment process for the tested steel.

## References

- [1] Bhadeshia H.K.D.H., *The first bulk nanostructured metal*, Science and Technology of Advanced Materials, nr 14, 2013.
- [2] Pytel S.M., Garcia C.I., DeArdo A.J., *Fracture toughness of ultra-low carbon bainitic steels for heavy plate applications*, Proceedings of International Conference on Processing Microstructure and properties of Microalloyed steels, Pittsburgh 1991.
- [3] Marcisz J., Burian W., Adameczyk M., *Właściwości mechaniczne stali maraging MS300 po starzeniu krótkotrwałym*, Prace Instytutu Metalurgii Żelaza nr 2, Gliwice 2013.
- [4] Burian W., Marcisz J., *Kinetyka procesów wydzieleniowych w stalach maraging podczas krótkotrwałego starzenia*, Prace Instytutu Metalurgii Żelaza nr 2, Gliwice 2013.
- [5] *Stale stopowe o wysokiej wytrzymałości do pracy w zakresie temperatur od  $-90$  do  $500^\circ\text{C}$* , broszura informacyjna o programie produkcyjnym Huta Baildon – Hutniczy Zakład Wytwórczo-Doświadczalnym „Mikrohuta”, KAW Katowice 1986.
- [6] Mahmoudi A., Zamanzad Ghavidel M.R., Hossein Nedjad S., Heidarzadeh A., *Aging behavior and mechanical properties of maraging steels in the presence of submicrocrystalline Laves phase particles*, Materials Characterization, ISSN 10445803 nr 10 Elsevier Science 2011.



- [7] Kladaric I., Krumes D., Marković R., *The Influence of multiple-solution annealing on kinetics of structural transformation of maraging steels*, Materials and Manufacturing Processes, issue 8, 2006.
- [8] Kawagoishi N., Nagano T., Moriyama M., Kondo E., *Improvement of fatigue strength of maraging steel by shot peening*, Materials and Manufacturing Processes issue 12, 2009.
- [9] Kladaric I., Kozak D., Krumes D., *The Effect of Aging Parameters on Properties of Maraging Steel*, Materials and Manufacturing Processes, issue 7–8, May 2009.
- [10] Mateja P., *Badania przemian fazowych w stalach maraging metodami rentgenowskimi*, U.Ś., Katowice 1990.
- [11] Ciszewski B., Przetakiewicz W., *Nowoczesne materiały w technice*, Bellona, Warszawa 1993.



