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SHORT-TIME AGEING OF MS350 MARAGING STEEL WITH AND WITHOUT PLASTIC DEFORMATION**KRÓTKOTRWALE STARZENIE STALI MARAGING MS350 W WARUNKACH ODKSZTAŁCENIA PLASTYCZNEGO**

The age hardening behaviour of MS350 maraging steel both in undeformed specimens and in specimens deformed immediately after ageing was studied. Experiments of short-time (15, 30 and 60 s) ageing at temperature up to 600°C were performed. It was found that after ageing at 480-500°C for 15-60 s without deformation the hardness reached the 450-525 HV. The time of ageing at 600°C to reach hardness 600 HV was 15 seconds. No precipitation in specimen aged at 500°C for 60 s was found in TEM and HRTEM investigations. Deformation immediately after ageing intensifies the process of hardening caused by temporary increase of temperature and increase of dislocation density. The hardness of specimens aged for 30 s and then deformed at 480-550°C was similar to the hardness at peak aged conditions (480°C/4h) and reached 600-630 HV. Material produced in industrial trials of cold forward flow forming and following laboratory ageing was analyzed. Increase of hardness from 370 HV to 590-630 HV for specimens taken from thin walled flow formed tube as a result of ageing at 460-490°C for 30 minutes occurred.

Keywords: maraging steel, short-time ageing, flow forming, precipitation kinetics, HRTEM

Przedstawiono wyniki badań procesu starzenia stali maraging w gatunku MS350 dla próbek nieodkształconych i odkształconych bezpośrednio po starzeniu. Przeprowadzono eksperymenty starzenia krótkotrwałego (15, 30 i 60 s) w temperaturze do 600°C. Stwierdzono, że po starzeniu w zakresie temperatury 480-500°C w czasie 15-60 s bez odkształcenia, twardość osiągnęła 450-525 HV. Czas starzenia w temperaturze 600°C do osiągnięcia twardości 600 HV wyniósł 15 s. Nie stwierdzono wydzielenia w próbkach starzonych w temperaturze 500°C przez 60 s w badaniach z zastosowaniem wysokorozdzielczego transmisyjnego mikroskopu elektronowego. Odształcenie bezpośrednio po starzeniu intensyfikuje proces umacniania wydzieleniowego w skutek chwilowego wzrostu temperatury oraz wzrostu gęstości dyslokacji. Twardość próbek starzonych przez 30 s i odkształconych w zakresie temperatury 480- 500°C była zbliżona do twardości uzyskiwanej dla warunków starzenia w celu uzyskania twardości maksymalnej (480°C/4godz.) i wynosiła 600-630 HV. Przeprowadzono badania materiału wytwarzanego w próbach przemysłowych zgniatania obrotowego i starzonego w warunkach laboratoryjnych. Dla próbek pobranych z rury cienkościennej uzyskano wzrost twardości z 370 HV do 590-630 HV w wyniku starzenia w zakresie temperatury 460-490°C przez 30 minut.

1. Introduction

Maraging steels belong to a special class of carbonless ultra-high-strength martensitic steels. A dominant mechanism of maraging steel strengthening is the precipitation of inter-metallic phases particles of the size of nanometres. The steels are suitable for engine components, such as crankshafts and gears, and the firing pins of automatic weapons that cycle from hot to cool repeatedly under high load. Their uniform expansion and easy machinability before ageing make maraging steel useful in wear resistant components of production lines and dies. Maraging steel's strength and ductility prior to ageing allows it to be formed into thin-walled products with the use of forward flow forming technology, reducing weight for a given strength. The forward flow forming saves raw material and reduces the production process time. Work hardening effect on solution-treated maraging steel with the use of flow forming is negligible. During flow forming process spray cooling of material is required because of possibility of the

occurrence of age hardening as a result of temporary increase of temperature caused by high amount of deformation at high strain rate.

The age hardening behaviour of MS350 maraging steel during short-time (15, 30 and 60 s) ageing at the temperature in the range 200-600°C without deformation and at the temperature in the range 20-550°C with deformation was investigated. The effect of short-time ageing and deformation on age hardening kinetics was described. Fragments of ageing curves at temperature up to 600°C and for time up to 60 seconds for MS350 steel were determined. The curves were obtained based on hardness measurements of samples after solution, heat treatment followed by ageing with and without deformation. Laboratory physical simulations were performed according to some parameters of industrial technology of cold forward flow forming of products made of MS350 maraging steel. Based on the results of laboratory investigation some parameters of flow forming industrial technology were modified.

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2. Literature review

Research on precipitate formation has received great attention due to its importance to the properties of maraging steels. Kinetics of precipitation in different maraging steels are not fully understood. The ageing behaviour of the maraging steels has been extensively studied [1–8], but especially early stages of ageing have not been precisely described. The fact that the composition of the hardening particles changes during ageing makes the determination of precipitation type and fraction even more difficult. Moreover, it should be noted that there is yet lack of fundamental theory explaining the processes just before precipitation.

Unless marked otherwise, the alloy composition in this paper is given in wt. %. Results available in the literature show different description of precipitation kinetics of maraging steels. Tewari et al. [1] studied the evolution of precipitates in maraging steel of grade 350 with the use of the complementary techniques of small angle X-ray scattering (SAXS) and transmission electron microscopy (TEM). Their results indicated towards the possibility of existence of two separate time-temperature-transformation (TTT) curves, one for the evolution of ω -phase and another for nucleation and growth of $\text{Ni}_3(\text{Ti},\text{Mo})$ -phase. Tewari et al. calculated the time of beginning of precipitation of $\text{Ni}_3(\text{Ti},\text{Mo})$ phase in 350 maraging steel (18Ni-12.5Co-4.2Mo-1.7Ti) at the temperature of ageing 510°C of the order of 30 minutes. In temperatures below 450°C, the time of beginning of precipitation of $\text{Ni}_3(\text{Ti},\text{Mo})$ phase is longer and amounts to several dozen hours on the average. Guo et al. [2] determined the time of start of precipitation in C250 maraging steel (18Ni-8Co-5Mo-0.5Ti) with the use of differential scanning calorimetry. They found that during ageing at 482°C the time of start of precipitation is about 30 s and the time of end of precipitation process is 1 hour. Specimen aged at 482°C for 1 hour reached hardness about 500 HV (hardness after solution heat treatment was 300 HV). Pardal et al. [3] investigated precipitation behaviour of 300 maraging steel (17.9Ni-9.4Co-4.8Mo-0.8Ti) solution treated and aged in the range of temperature 440–650°C. They pointed out that after ageing at 560°C for about 1 hour the hardness reached 600 HV. Sha [4] shows that the early stage of precipitation hardening can be describe using the Johnson-Mehl-Avrami equation. He found that in maraging steel C-300 (18.5Ni-9Co-4.8Mo-0.6Ti) aged at 420°C for period of time between 160 seconds and 25 minutes hardness increased by about 100 HV. Li and Yin [5] examined 18Ni(350) maraging steel (18Ni-12Co-4.5Mo-1.3Ti) aged at 500°C for 2, 5, 15, 30 and 60 minutes. The authors found that the precipitation process is preceded by the redistribution of atoms, which results in the formation of Fe-Co-rich areas and Ni-Mo-Ti-rich areas. The redistribution of atoms is fast at the initial stages of ageing and then it is slow. Intermetallic compounds precipitate on the Ni-Mo-Ti-rich areas. Rajkumara et al. found that after ageing at 482°C for 15 minutes the hardness increased by about 150 VHN (from 360 to 510 VHN) [6]. The hardness

of the M250 maraging steel (17.89Ni-8.16Co-4.88Mo-0.43Ti) increased with ageing time peaked at intermediate ageing period followed by continuous decrease at longer durations of ageing [6]. The initial increase in the hardness up to 3 hours is attributed to the precipitation of Ni_3Ti intermetallic precipitates from the martensite matrix as resulted from TEM studies. The continuous increase in the hardness at intermediate periods (3–40 hours) is attributed to additional precipitation of fine Fe_2Mo from the solid solution. Changes in the solute distribution as well as the evolution of precipitation, microstructure and mechanical properties were studied in an experimental Fe-20Ni-1.8Mn-1.5Ti-0.59Al alloy during ageing at 550°C [7]. An initial hardening reaction within 5 s was reported, which was remarkable in terms of extent and rapidity. Within 5 s at 550°C, the hardness increased by 200 VHN and the yield strength increased by 215 MPa to more than 900 MPa. This strengthening was caused by the formation of complex multi-component atomic co-clusters containing primarily Ni-Ti-Al-Mn. After 60 s ageing, the appearance of discrete precipitation of needle-shaped $\gamma\text{-Ni}_3\text{Ti}$ particles was associated with a second rise in hardness towards an eventual peak at 600 s. Shekhter et al. [8] studied ageing sequence in Fe-25.3Ni-1.7Ti alloy undeformed and cold deformed prior to ageing. They found an excellent combination of hardness, strength, and ductility after only 5 seconds ageing at 550°C. They proposed that this rapid strengthening is due to a dislocation friction effect arising from the formation of fine Ni-Ti atomic co-clusters during short ageing time.

3. Experimental procedures

Material. Heat of MS350 maraging steel (Fe-18Ni-12Co-4Mo-Ti) was made in high-vacuum induction furnace having melting pot of capacity of 100 kg and cast in vacuum to water cooled copper mould. Ingot of cross section of 100×100 mm was subject to homogenizing heat treatment (1250°C/24 hours) followed by mechanical surface skinning. Subsequently, hot working was carried out in order to obtain material in form of round bars in diameter of 80 mm and flat bars with cross section 20×25 mm, the samples of which were prepared. The bars were solution treated at 850°C for 30 minutes followed by cold water quenching. Following solution treatment, samples assigned for heat treatment and/or for compression tests were prepared to obtain dimension $\phi 8 \times 10$ mm and $\phi 6 \times 8$ mm. Round bars in diameter of 80 mm were used for production of die stampings. The stampings were used for two passes forward flow forming of tubes with the first wall thickness reduction from 3.8 mm to 2.1 mm and the second from 2.1 mm to 1.1 mm without any intermediate annealing. The thin-walled tubes made of MS350 steel in industrial trials of cold flow forming were cut into small samples, which were aged at 460°C, 480°C and 490°C for various durations ranging from 30 minutes to 8 hours, and air cooled. Chemical composition of the examined steel is presented in Table 1.

TABLE 1

Chemical composition of the MS350 maraging steel (N18K12M4T), wt. %

| C | Mn | Si | P | S | Cr | Ni | Co | Mo | Ti | Cu | Al |
|--------|-------|-------|------|------|-------|------|------|------|------|-------|------|
| <0.005 | <0.05 | <0.05 | 0.01 | 0.01 | <0.01 | 17.5 | 12.1 | 4.10 | 1.90 | <0.05 | 0.11 |

Experimental methods. Laboratory experiments of short-time ageing with and without deformation using the Gleeble 3800 simulator were carried out. Cylindrical samples were aged at the range of temperature from 200 to 600°C for 15, 30 and 60 seconds. Table 2 shows parameters of heat treatment of maraging steel without deformation. Figure 1 shows examples of temperature changes on the surface of specimens during heat treatment in Gleeble simulator. Total time of ageing consists of three stages (time of heating at the rate of 10 K/s; annealing time and cooling time) and depends on temperature of heat treatment. Uni-axial compression tests after ageing at temperature range 20-550°C for 30 seconds were carried out. Compression tests were performed at the ageing temperature and at the strain rates of 1 s⁻¹ and 10 s⁻¹. One step deformation of value from 1.4 to 1.6 was conducted. Parameters of experiments for specimens deformed after ageing for 30 seconds are listed in Table 3. Figs. 2 and 3 show examples of temperature changes of specimens deformed after ageing. Temperature of specimens during experiments of ageing and compression was measured with the use of thermocouples. Fig. 2 shows examples of temperature changes on the surface of specimens during deformation at 400°C. As it is shown

TABLE 2
Parameters of ageing without deformation carried out with the use of Gleeble 3800 simulator

| Ageing temperature, °C | Ageing time, s |
|------------------------|----------------|
| 350 | 15 |
| 400 | |
| 450 | |
| 480 | |
| 500 | |
| 550 | |
| 600 | 30 |
| 200 | |
| 300 | |
| 350 | |
| 400 | |
| 450 | |
| 480 | 60 |
| 490 | |
| 500 | |
| 550 | |
| 600 | |
| 350 | |
| 400 | |
| 450 | |
| 480 | |
| 500 | |
| 550 | |
| 600 | |

in Fig. 2, after the beginning of compression the surface temperature increases, reaches the maximum and then decreases below predicted temperature. In fact, the process of deformation proceeds in the range of temperature depending on predicted temperature and parameters of compression (strain rate, amount of deformation).

Results of temperature measurements on the surface of specimens during compression tests are shown in Table 5. Fig. 3 shows an example of temperature changes on the surface and in the middle of specimen deformed at 400°C with the strain rate of 10 s⁻¹. Maximum temperature of about 640°C in the middle of the specimen for predicted temperature of 400°C was detected. The experiment was conducted with thermocouple mounted inside the deformed specimen during overall compression.

For all specimens examined the hardness measurements were performed. Fig. 4 presents the scheme of hardness measurements of deformed specimens. Points IV-VI of hardness measurements represent area of specimens corresponding to real value of obtained deformation [9]. Examinations of microstructure of specimens after heat treatment and after heat treatment and followed deformation using light microscope (LM) and transmission electron microscope (TEM) were carried out. Specimens for LM examination were etched using 5% nital. Thin foils for TEM were prepared by twin jet polishing using 5 pct perchloric acid in methanol at -30°C. High resolutions transmission electron microscopy technique (HRTEM) was used to examine early stages of precipitation and/or atoms redistribution in the matrix. Specimens were examined in ultra-high resolution (S/TEM) -transmission electron microscope TITAN 80-300.

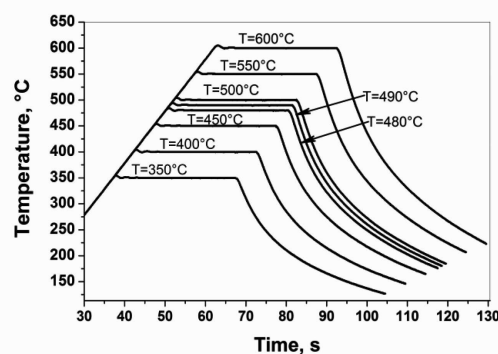


Fig. 1. Temperature changes on the surface of the specimens for experiments with ageing time 30 s without deformation

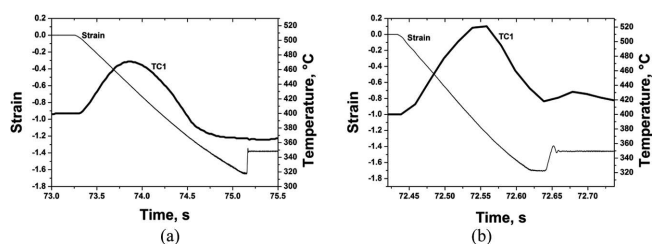


Fig. 2. Changes of temperature on the surface (TC1) of specimen during compression with the strain rate of 1 s⁻¹ (a) and 10 s⁻¹ (b) at 400°C after ageing for 30 s

TABLE 3

Parameters of ageing and deformation realized by one step uni-axial compression with the use of Gleeble 3800 simulator (time of ageing before compression was 30 s)

| Temperature of ageing and deformation, °C | Strain ϵ | Strain rate, s^{-1} |
|---|-------------------|-----------------------|
| 550 | 1.4-1.6 | 1 |
| 500 | | |
| 480 | | |
| 450 | | |
| 400 | | |
| 350 | | |
| 300 | | |
| 200 | | |
| 100 | | |
| 20 | | |
| 400 | 1.4-1.6 | 10 |
| 350 | | |
| 300 | | |
| 200 | | |
| 100 | | |
| 20 | | |

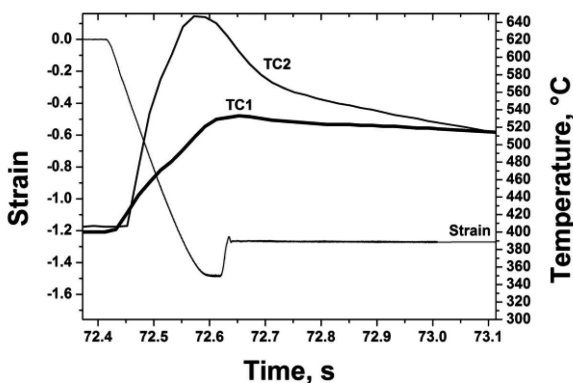


Fig. 3. Changes of temperature on the surface (TC1) and in the middle (TC2) of the specimen during compression with the strain rate of $10 s^{-1}$ at $400^{\circ}C$ after ageing for 30 s

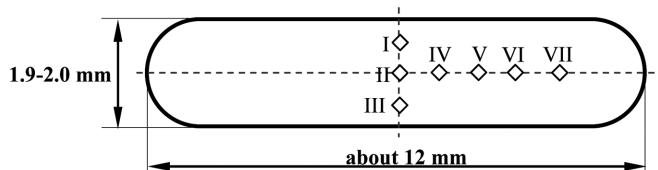


Fig. 4. Scheme of hardness measurements of aged and deformed specimens

4. Results and discussion

Laboratory physical simulation. Results of hardness measurements of specimens after short-time ageing at temperature in the range of $200-600^{\circ}C$ are shown in Fig. 5 and in Table 4. Steel hardness after solution heat treatment was 340 HV. The first noticeable increase in hardness to 380 HV took place after annealing at the temperature of $400^{\circ}C$ for 15 s. Specimens aged at standard temperature of heat treatment used for MS350 steels ($480-500^{\circ}C$) for 15-60 s reached hardness in the range of 450-525 HV. Ageing at temperature $480^{\circ}C$ for 60 s causes the hardness increase by about 170 HV (hardness reaches 511 HV). The time of ageing at $600^{\circ}C$ to reach hardness 600 HV was only 15 seconds.

TABLE 4

Results of hardness measurements of specimens after solution heat treatment and short-time ageing at the temperature range $200-600^{\circ}C$

| Hardness after solution heat treatment | 340 HV10 | | 34 HRC | | | |
|--|---|-----|---|-----|---|-----|
| | Hardness after 15 s ageing (mean value) | | Hardness after 30 s ageing (mean value) | | Hardness after 60 s ageing (mean value) | |
| | HV10 | HRC | HV10 | HRC | HV10 | HRC |
| 200 | – | – | 336 | 34 | – | – |
| 300 | – | – | 350 | 36 | – | – |
| 350 | 363 | 37 | 357 | 37 | 373 | 37 |
| 400 | 380 | 39 | 375 | 39 | 397 | 41 |
| 450 | 419 | 41 | 423 | 44 | 446 | 44 |
| 480 | 454 | 45 | 454 | 46 | 511 | 49 |
| 490 | – | – | 471 | 48 | – | – |
| 500 | 477 | 47 | 489 | 49 | 525 | 49 |
| 550 | 555 | 52 | 550 | 52 | 592 | 52 |
| 600 | 605 | 54 | 581 | 53 | 599 | 54 |

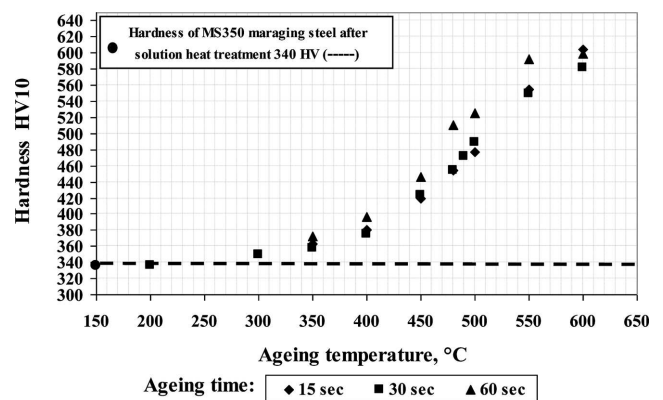


Fig. 5. Hardness changes after short-time ageing of MS350 maraging steel at $200-600^{\circ}C$

The microstructure of MS350 maraging steel after short-time ageing is shown in Fig. 6. Prior-austenite grain boundaries after ageing at $500^{\circ}C$ for 15 seconds were difficult to etch (Fig. 6a) similarly to the material in solution heat

treatment condition. The microstructure of specimens aged at 500°C for 30 s and 60 s and at 600°C for 30 s consisted of packets of martensite, within prior-austenite grains (Fig. 6b-d). The austenite grains, which had transformed into packets of martensite, could still be recognised due to the preferential etching along their boundaries and also due to the fact that the martensite packets within an austenite grain did not extend beyond the respective prior-austenite grain.

TEM images of the samples aged at 500°C for 60 s are shown in Fig. 7. The bright-field image in Fig. 7a shows the microstructure comprising of martensite laths with tangled dislocations within. No precipitation was found at magnification up to 150 000 \times in TEM examination.

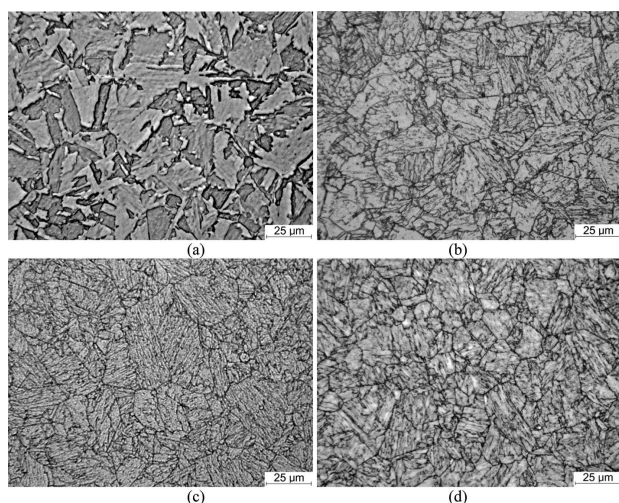


Fig. 6. Microstructure of specimens after ageing and without following deformation: a) at 500°C for 15 seconds; b) at 500°C for 30 seconds; c) at 500°C for 60 seconds; d) at 600°C for 30 seconds

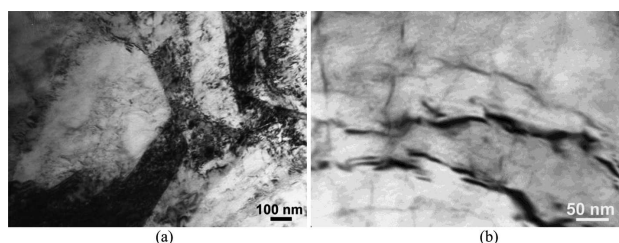


Fig. 7. Bright-field transmission electron microscope images of specimen aged at 500°C for 60 seconds (hardness 525 HV)

A HRTEM investigation shows that the matrix consists only of $\text{Fe}\alpha$ planes with local discontinuity indicating dislocations and clusters of atoms (Fig. 8). No precipitation in specimen aged at 500°C for 60 s was found in HRTEM investigation. Hardness increase in the early stages of ageing could have been caused only by redistribution of atoms forming precipitations in the beginning of ageing. Microstructure investigation results and hardness measurements indicate towards the possibility of existence of high level of stresses in the matrix due to only redistribution of the atoms.

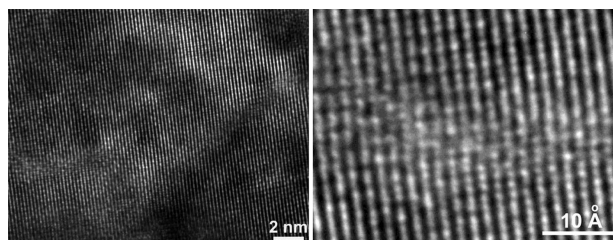


Fig. 8. HRTEM images of specimen aged at 500°C for 60 seconds

Results of hardness measurements of specimens short-time aged and compressed at temperature range 20-550°C are shown in Table 5. Fig. 9 shows the hardness changes resulting from ageing for 30 s and from deformation. Fig. 9 also shows the hardness of specimens aged for 30 s without deformation as a reference to compressed material. Results of measurement of hardness of specimens compressed at room temperature show the strain hardening effect in MS350 steel of about 40-70 HV (about 15%). At the temperature range of ageing and deformation from 100°C to 400°C monotonic increase of hardness from 390 to 500 HV for the strain rate of 1 s⁻¹ and from 430 to 500 HV for the strain rate of 10 s⁻¹ was found. In specimens aged for 30 s and then compressed at temperatures up to 300°C no cracks were found. First visible cracks were observed on the side surface of specimens aged for 30 s and compressed at 350°C when hardness reached about 500 HV. At temperature above 350°C specimens cracked during compression which made further uniform deformation impossible. Due to ageing at the temperature of 480°C for 30 s and following compression the hardness increases by about 250 HV and reaches 590 HV.

Hardness of specimens aged for 30 s at temperature range 200-550°C and then deformed in comparison with specimens aged in the same conditions without deformation is higher by about 120-160 HV depending on the temperature of ageing and strain rate. Maximum hardness increase due to deformation (160 HV) occurred in specimens aged and compressed at 450°C with the strain rate of 1 s⁻¹. The smaller strain rate the longer the time for precipitation and/or atoms redistribution during and after deformation at ageing temperature. Slower strain rate, by making enough time available for redistribution of solutes, leads to partitioning of the matrix into Ni-rich and Ni-depleted regions. Hardness increase of specimens compressed after ageing resulted from strain hardening and precipitation and/or atoms redistribution hardening processes. Moreover, these strengthening processes proceed simultaneously and plastic deformation strongly intensifies precipitation. The additional effect of hardening resulted from temporary increasing of temperature due to deformation.

Examples of light micrographs of the material aged and then deformed in Gleeble simulator are shown in Fig. 10. Examination of microstructure was performed in the area of hardness measurement points. Specimens put to the uni-axial compression tests were characterized by non-uniform microstructure on the cross-section. The microstructure consists of grains (sub-grains) strongly elongated in the direction of plastic flow. The prior-austenite grain boundaries in deformed material were not visible. The type of microstructure in deformed areas indicates good flowability of the material.

TABLE 5

Results of hardness measurement of aged and then deformed specimens (obtained value of deformation was about 1.4-1.6)

| Temperature of ageing and deformation, °C | Range of surface temperature during deformation, °C | Strain rate, s ⁻¹ | Hardness HV10 | | | | | | |
|---|---|------------------------------|-----------------------------|-----|-----|-----|-----|-----|-----|
| | | | Measurement points (Fig. 4) | | | | | | |
| | | | I | II | III | IV | V | VI | VII |
| 550 | 527-611 | 1 | 639 | 631 | 660 | 628 | 630 | 615 | 640 |
| 500 | 483-595 | | 561 | 544 | 605 | 613 | 622 | 603 | 598 |
| 480 | 480-575 | | 561 | 577 | 579 | 589 | 591 | 601 | 588 |
| 450 | 450-529 | | 558 | 573 | 596 | 590 | 591 | 579 | 567 |
| 400 | 366-471 | | 488 | 508 | 514 | 514 | 503 | 514 | 508 |
| | 358-465 | | 473 | 493 | 493 | 483 | 483 | 478 | 493 |
| 350 | 372-444 | | 481 | 494 | 451 | 478 | 475 | 483 | 490 |
| 300 | 273-375 | | 417 | 437 | 425 | 437 | 441 | 441 | 425 |
| | 274-375 | | 425 | 429 | 425 | 437 | 433 | 433 | 433 |
| 200 | 191-289 | | 394 | 401 | 397 | 405 | 401 | 405 | 401 |
| | 195-290 | | 397 | 405 | 390 | 409 | 409 | 409 | 405 |
| 100 | 100-203 | | 383 | 401 | 383 | 397 | 383 | 401 | 380 |
| | 100-199 | | 383 | 397 | 383 | 397 | 394 | 383 | 373 |
| 20 | 20-137 | | 376 | 380 | 380 | 380 | 387 | 401 | 390 |
| | 20-144 | 373 | 387 | 383 | 387 | 387 | 383 | 373 | |
| 400 | 400-520 | 488 | 488 | 488 | 493 | 498 | 498 | 483 | |
| | 396-513 | 483 | 483 | 483 | 493 | 498 | 503 | 473 | |
| 350 | 350-495 | 489 | 490 | 469 | 479 | 478 | 488 | 510 | |
| 300 | 300-458 | 464 | 483 | 464 | 478 | 473 | 483 | 483 | |
| | 300-454 | 454 | 488 | 464 | 478 | 468 | 468 | 468 | |
| 200 | 200-356 | 429 | 454 | 433 | 454 | 454 | 464 | 446 | |
| | 200-351 | 409 | 446 | 433 | 464 | 450 | 450 | 437 | |
| 100 | 100-279 | 405 | 441 | 409 | 433 | 433 | 429 | 425 | |
| | 100-295 | 401 | 425 | 409 | 425 | 409 | 420 | 420 | |
| 20 | 20-209 | 390 | 413 | 401 | 397 | 401 | 409 | 390 | |
| | 20-204 | 405 | 420 | 401 | 413 | 409 | 425 | 420 | |

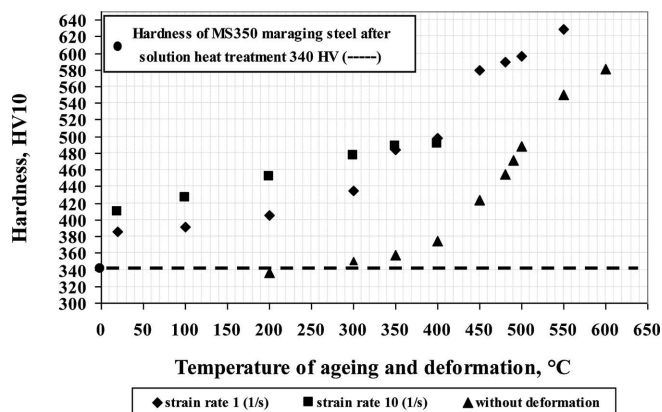


Fig. 9. Hardness changes of specimens aged and then deformed in Gleeble simulator at temperature range 20-550°C and aged without deformation at temperature range 200-600°C. Ageing time for all experiments was 30 s

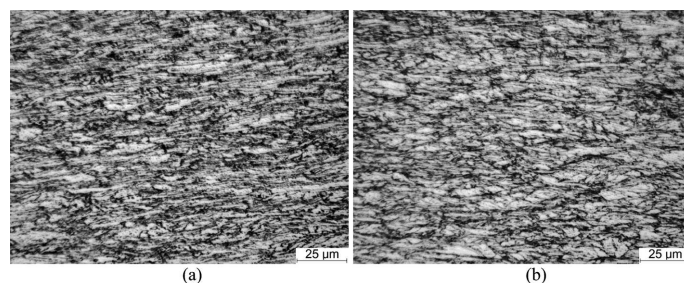


Fig. 10. Microstructure in the middle of aged and deformed specimens. Temperature of deformation 400°C, strain rate (a) 1 s⁻¹, (b) 10 s⁻¹

Examination of material after industrial trials of flow forming. Specimens taken from thin walled cold flow formed tube were aged at 460, 480 and 490°C for period of time from 0.5 to 8 hours. Hardness of steel after solution treatment was about 320 HV. During cold flow forming the hardness

in examined areas increased to 370-420 HV as an effect of strain hardening and/or age hardening. For further laboratory experiments of heat treatment specimens of equal hardness (370 HV) were chosen.

Results of hardness measurements of aged specimens are shown in Fig. 11. Increase of hardness from 370 to 590-630 HV for time of heat treatment of 0.5 hour at ageing temperature in the range 460-490°C occurred. Maximum hardness of 670 HV for specimen aged at 490°C for 1 hour was found. Fig. 12 shows the microstructure of specimen cold flow formed and then aged at 480°C for 1 hour. Microstructure was characterized by grains and/or subgrains elongated in the direction of plastic flow.

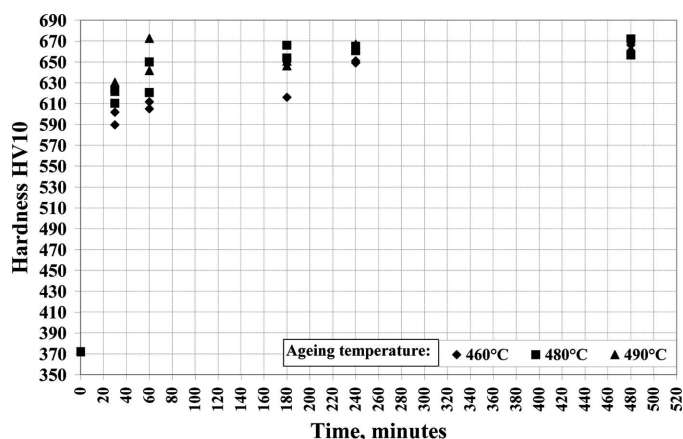


Fig. 11. Hardness vs. ageing time for temperature of ageing: 460°C, 480°C and 490°C. Specimens from thin walled cold forward flow formed tube

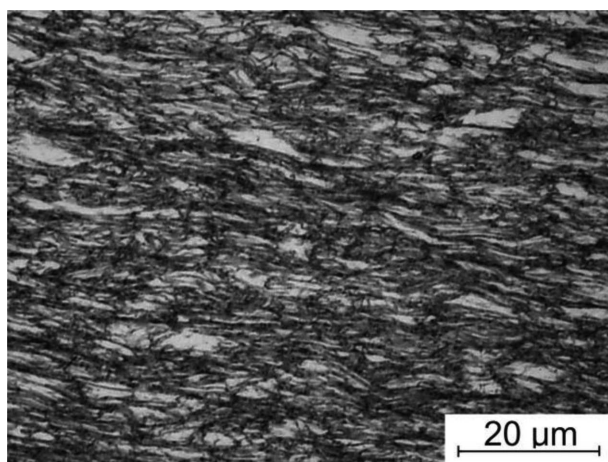


Fig. 12. Microstructure of specimen after cold flow forming and following ageing at 480°C for 1 hour (hardness 650 HV). Near-to-surface layer on the side of spinning rolls

5. Summary and Conclusions

The short-time age hardening behaviour of MS350 maraging steel both in undeformed specimens and in specimens deformed immediately after ageing was examined. The first noticeable increase of hardness for specimens aged at 400°C for only 15 s without deformation was observed. It

was found that during ageing at 480-500°C for 60 s the hardness increased by about 170-180 HV and reached 510-525 HV. Hardness of specimens aged at 600°C for 15 s without deformation reached 600 HV. No precipitation was found in specimen aged at 500°C for 60 s in HRTEM investigation. The hardness increase resulted from redistributions of atoms of elements before precipitation. The process of atoms redistribution produces relatively high stresses in the matrix of maraging steel and then extra energy is required for dislocations to cut through the clusters.

The beginning of matrix hardening for specimens aged at the temperature of 200°C for 30 s and then deformed was observed. The effect of strain hardening of solution treated MS350 maraging steel at room temperature is about 10-15%. After ageing for 30 s and following deformation at 480-500°C the hardness reached 580-620 HV (70-80%). This is a similar level of hardness for this grade of steel to hardness at peak aged condition (480°C/4h). The hardness increase in specimens aged and then deformed could result from intensification of precipitation process caused by temporary temperature increase during and after deformation and increase of dislocation density. Increase of dislocations density has an influence on number of nuclei of precipitation and on negligible strain hardening of matrix. Moreover, the higher density of dislocations the easier the diffusion of elements in the steel matrix.

Material after industrial trials of cold forward flow forming and following ageing was examined. Increase of hardness from 370 HV to 590-630 HV for specimens taken from thin-walled flow formed tube after ageing at 460-490°C for 30 minutes occurred. It was found that maximum hardness of specimen of about 670 HV occurred after plastic deformation by cold flow forming and ageing at 490°C for 60 minutes. Parameters of final heat treatment of thin-walled tubes could be modified on the basis of examination results.

Further research is oriented towards determination of properties of maraging steels after short-time ageing, including their stability (e.g. thermal stability) and possibility of application in this condition.

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