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POLSKA AKADEMIA NAUK

A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 58 2013 Issue 2
DOI: 10.2478/amm-2013-0038

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THE MICROSTRUCTURES AND HARDNESS ANALYSIS OF A NEW HYPEREUTECTOID Mn-Cr-Mo-V STEEL

ANALIZA MIKROSTRUKTURY I TWARDOŚCI NOWEJ, NADEUTEKTOIDALNEJ STALI Mn-Cr-Mo-V

The results of a microstructure and hardness investigations of a new hypereutectoid Mn-Cr-Mo-V steel, imitating by its chemical composition tool steels, are presented in the paper. The microstructure as well hardness changes, caused by austenitising and tempering temperatures were assessed, for samples quenched and sub-quenched in liquid nitrogen, directly after the quenching treatment. Additionally, the influence of the tempering temperature on the volume fraction of the retained austenite was estimated. New hypereutectoid steel, after an appropriate heat treatment obtained the relevant hardness of the tools used in the cold and hot working proces.

It was indicated that the steel hardness increases with the increases of the austenitising temperature. At 800°C the hardness of the quenched samples were equal 895HV, and for the sub-quenched samples 937HV. The maximum hardness, after tempering (746HV), was found at a temperature of 520°C.

It will be possible, in future, to apply this obtained investigation results in designing chemical compositions and microstructures of the new hypereutectoid alloyed steels of properties required by their users.

Keywords: microstructure, hardness, quenching, tempering, hypereutectoid steel

W artykule zamieszczono wyniki badań mikrostruktury i twardości nowej, nadeutektoidalnej stali Mn-Cr-Mo-V imitującej składem chemicznym stale narzędziowe. Na próbkach nowej stali niewymrożonych oraz wymrożonych w ciekłym azocie bezpośrednio po zabiegu hartowania oceniono zmiany zachodzące w jej mikrostrukturze oraz zmiany twardości z temperaturą austenityzowania i z temperaturą odpuszczania. Dodatkowo, oceniono zmianę udziału objętościowego austenitu szczątkowego w badanej stali z temperaturą odpuszczania. Nowa stal nadeutektoidalna, po zastosowaniu odpowiedniej obróbki cieplnej uzyskuje twardości oczekiwane przez użytkowników narzędzi stosowanych w przeróbce plastycznej na zimno i na gorąco. Wykazano, że ze wzrostem temperatury austenityzowania jej twardość rośnie osiągając przy 800°C wartości: 895HV (dla próbek niewymrożonych) oraz 937HV (dla próbek wymrożonych). Natomiast maksymalną twardość po odpuszczaniu (746HV) odnotowano przy temperaturze 520°C. Uzyskane wyniki badań będzie można wykorzystać w przyszłości przy projektowaniu składów chemicznych i mikrostruktur nowych, nadeutektoidalnych stali stopowych o wymaganych przez użytkowników własnościach.

1. Introduction

In spite of a fast development of the modern material engineering and looking for new materials, hypereutectoid steels and hot work tool steels still belong to basic materials used for tools for shaping products by means of machining and plastic working, and for elements of measuring equipment [1÷4]. Unalloyed hypereutectoid steels are characterised by a low hardenability and their properties and applications depend mainly on a carbon content [1÷3, 5÷7]. The main application of these steels is for: punches, reamers, surgeons instruments, stonework tools, forging dies [1÷3, 8].

Whereas alloyed hypereutectoid steels are characterised by a much better hardenability, ensuring the proper thickness of a martensitic layer and core strength, and by the high hardness (usually above 60HRC) – at simultaneous sufficient ductility, good abrasion and thermal resistance [1÷3, 5, 9]. These properties are obtained by steels due to the proper balancing of their chemical composition, i.e. carbon and carbide forming elements content (first of all: Cr, Mo, W and V) and a properly developed and performed heat treatment technology [1÷3, 5, 7, 9÷14]. Such steels are applied for producing tools for plastic working and cutting, i.e. for punching dies, drawing dies, drawing rings and milling cutters [1÷3, 8].

The chemical composition of the new hypereutectoid alloyed steel was designed to have possibility to perform in the laboratories of the Department of Physical and Powder Metallurgy, AGH technical heat treatment tests [9]. The chemical composition of this steel was designed considering its application for tools with high hardness used in cold and hot working processes. The critical temperatures of this new steel were determined. The microstructure at the annealed state was assessed and the selected mechanical properties, it means strength and plastic indices in the static tensile test and ductili-

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ty in the impact test, were evaluated [9]. The mentioned results were obtained after quenching from an 800°C austenitising temperature (Ac_{1k}+ 55°C). Thus, the results contained in the hereby paper are the continuation of the research presented in paper [9].

The hardness and microstructure changes after tempering are estimated in this paper. They were evaluated after its previous quenching from a temperature corresponding to the solubility limit of carbides and carbonitrides of vanadium in the austenite (1160°C, i.e. Acm + 110°C). On account of the height austenitising temperature (1160°C), the obtained results are of a scientific character and – for sure – will increase knowledge of microstructure and properties of the new hypereutectoid steels imitating tool steels by their chemical composition.

2. Research material

Investigations were performed on the specially designed model steel melt, which chemical composition is given in Table 1.

TABLE 1
Chemical composition of the investigated steel, % by mass

C	Mn	Si	P	S	Cr	Mo	V	Al	N
1.25	2.05	0.25	0.02	0.015	1.61	0.39	0.44	0.025	0.005

The tested steel (see Table 1) contains a high concentration of carbon (app. 1.25%) and alloying elements (mainly Mn and Cr). The vanadium concentration was selected at the level of 0.44%. At such concentrations of carbon, vanadium and the others alloying elements, carbides and carbonitrides of vanadium are fully dissolved in the austenite only above a temperature of 1160°C.

Tests, so-called quenching and tempering series on steel with higher vanadium content could not be performed regarding to the temperature range limitation of the lab furnace.

The steel melt was made in the Foundry Institute in Cracow, in the open induction furnace using Armco iron and ferroalloy Fe-V 75%. By means of the open die forging technology the rods of transverse dimensions 20×35 mm were made in Huta Batory, from ingots of a diameter of app. 80 mm and a mass of app. 50 kg.

3. Experimental procedure

The measured critical temperatures of the new steel are given in paper [9], respectively: Ac_{1p} =720°C, Ac_{1k} =745°C and Ac_m =1050°C. These temperatures are verified by the metallographic method by means of the observation of polished sections of samples from the so-called quenching series. This series was performed to investigate the influence of the austenitising temperature on the hardness of the steel. Samples of dimensions 20×15×10 mm were austenitised in the temperature range from 720 to 1200°C for 30 minutes and then water cooled. The second part of samples, directly after quenching, ware sub-quenched in liquid nitrogen for one hour.

In order to investigate the hardness changes of the steel with a tempering temperature the so-called tempering series were made for samples previously quenched as well as for samples quenched and sub-quenched in liquid nitrogen for one hour. The samples ware sub-quenched to decrease the volume fraction of the retained austenite and to increases the steel hardness after quenching. Before tempering, samples were quenched from a temperature of 1160°C, at which all carbides and carbonitrides of vanadium (MC type) were dissolved in the austenite. This meant dissolution of the total vanadium amount, which was shown by the chemical analysis of the investigated alloy. A temperature of 1160°C was determined by means of the thermodynamic model given in paper [15], and by using diagrams determining the occurrence range of the homogenous austenite in the Fe-C-V alloys – for various volume fractions of vanadium [16]. These methods were metallographically verified by the observation of polished sections quenched from higher and higher temperatures.

The tempering series was performed for the temperature range 100÷700°C. A special attention was directed towards the temperature range 500÷600°C, within which the tempering temperature was changed every 20°C. The tempering time was 2 hours. Each variant of the heat treatment was repeated for three samples. Treatments of annealing, austenitising and tempering were carried out in the laboratory furnace, RHF 16/19 type, of the Carbolite Company.

The retained austenite volume fraction was assessed in samples quenched in water from a temperature of 1160°C and tempered in the temperature range 100÷700°C. Measurements of the retained austenite volume fraction were made on samples of dimensions: 20×15×10 mm using the direct comparison method acc. to [17]. Notations in the X-ray diffractometer TUR-M61 were made from transverse metallographic microsections, polished mechanically.

Hardness measurements of quenched samples and of quenched and tempered samples were performed by the Vickers apparatus, type HPO 250, at the indenter load of 30 kG (294 N). In order to remove the decarburised layer during the heat treatment the tested surface was preliminarily mechanically grinded to a depth of app. 1 mm. The final grinding was done on abrasive papers of a gradually lowered grade of abrasive grains. Four hardness measurements were made for each sample and the arithmetic mean was calculated from the obtained results.

For the assessment of the new steel microstructure in the quenched state and in the quenched and tempered state, the optical microscope Nu of the Carl Zeiss Company from Jena was used. Samples for metallographic observations were grinded on the magnetic grinder with a borazon grinding wheel and then on abrasive papers. The preliminary polishing of the polished sections was done on a diamond paste and the final polishing by Al_2O_3 suspension. Polished sections were etched by 2% nital. The photographic documentation of microstructures was done at the magnification of app. 630 x.

4. Results and discussion

The austenitising temperature influence on the hardness of the new Mn-Cr-Mo-V steel samples, previously annealed and then quenched in water as well as quenched and sub-quenched in liquid nitrogen (for 1 hour), are presented in Figure 1.

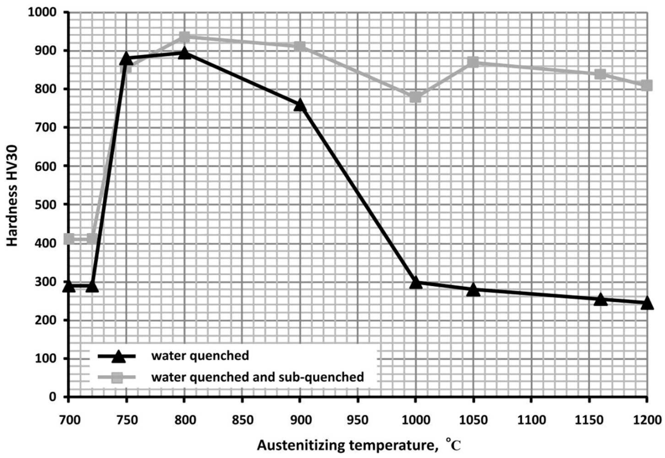


Fig. 1. Influence of the austenitising temperature on the hardness of the previously annealed and then quenched in water and of the quenched and sub-quenched in liquid nitrogen the new steel samples

As can be seen (Fig. 1) the austenite starts forming in the tested steel above 720°C, since only above this temperature the hardness of quenched samples starts increasing fast (both sub-quenched and quenched). The hardness maximum in these both cases was obtained after quenching samples from a temperature of 800°C. The hardness of quenched samples after water quenching was app. 895 HV30, while the hardness of samples sub-quenched in liquid nitrogen after water quenching was 937 HV30. In case of only quenched samples after exceeding the austenitising temperature, corresponding with the steel hardness maximum, its significant decrease is observed. This hardness decrease should be related to the simultaneous decrease of the martensite volume fraction and increase of the soft retained austenite volume fraction in the new steel microstructure. This austenite volume fraction, after quenching from a temperature of 1160°C, determined by the X-ray method, was equal 95 vol. % and its hardness was 249 HV.

In case of samples sub-quenched in liquid nitrogen, directly after the quenching treatment, within the whole austenitising temperature range significantly higher hardness values were obtained (especially within the temperature range 900÷1200°C). This is related to the transformation of the retained austenite – formed in larger and larger amounts at higher and higher temperatures – into the fresh martensite during the sub-quenching treatment. When the austenitising temperature is increased from 800 to 1200°C the hardness of the new steel samples quenched in water and then sub-quenched in liquid nitrogen decreases from 937 to 808 HV. The lower hardness of the sample quenched in water from a temperature of 1000°C and then sub-quenched (778 HV) can be the most probably related either to the sudden austenite grain growth or to the possibility of it's tempering during grinding.

Photographs of the selected microstructures of the new steel samples, austenitised in the temperature range 720÷1200°C and water cooled as well as austenitised in the same temperature range but sub-quenched in liquid nitrogen for 1 hour – directly after the quenching treatment – are shown in Figure 2.

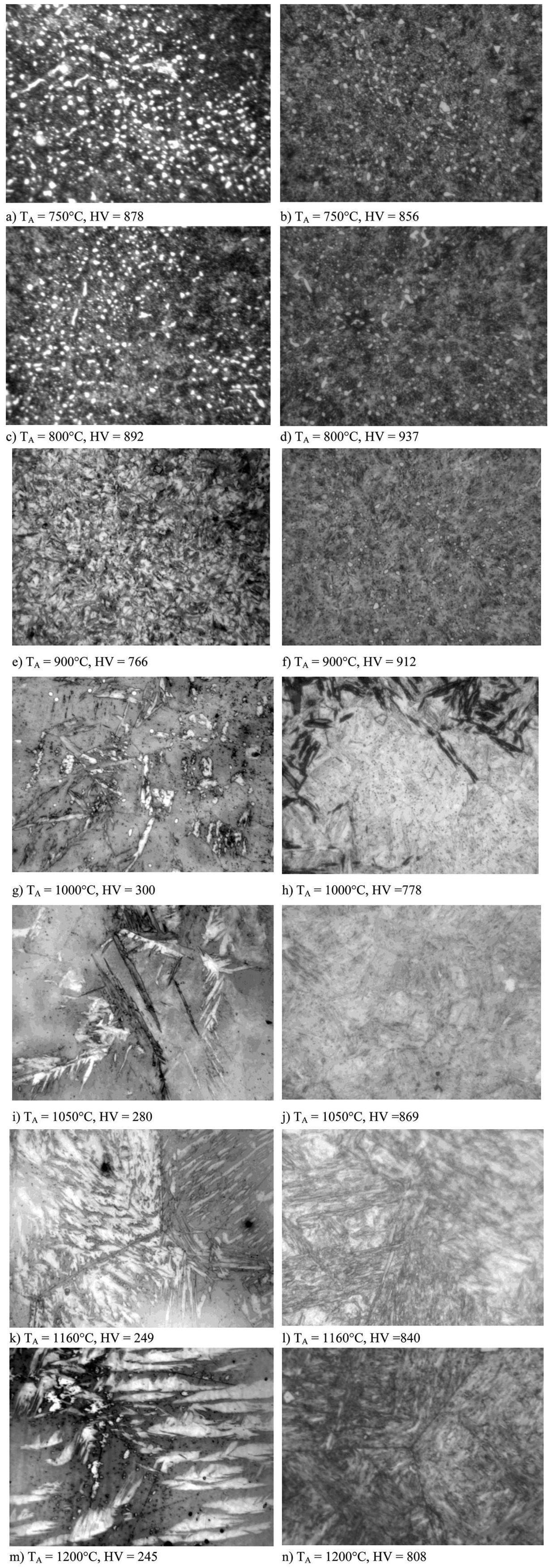


Fig. 2. Photographs of the selected microstructures of the tested alloy samples austenitised in the temperature range 750÷1200°C, without sub-quenching (left side – Fig. a, c, e, g, i, k, m) and with sub-quenching at – 196°C (right side – Fig. b, d, f, h, j, l, n), magnification 630x, etching: nital 2%

In the microstructure of samples water cooled from the austenitising temperatures – in the temperature range: 750÷800°C (Fig. 2a÷d) – fine-acicular martensite is seen, also a small volume fraction of the retained austenite and undissolved (during austenitising) fine-dispersive carbides (the most probably) of vanadium of the MC type, and of other elements (e.g. chromium). Beginning from the austenitising temperature of 900°C (Fig. 2e) the retained austenite starts playing a significant role and a large volume fraction of fine-dispersive undissolved vanadium carbides, MC type, is clearly seen in the microstructure. Within the temperature range: 1000÷1200°C (Fig. 2g÷n) the significant austenite grain growth of the new steel is seen. A small volume fraction of the coarse-acicular martensite is seen in Fig. 2g, i, k, m, while the fresh martensite originated from the retained austenite transformation is seen in microstructures presented in Fig. 2h, j, l, n. The analysis of Figure 2k, l allows to state that a temperature of 1160°C corresponds with the total solubility of fine-dispersive carbides and carbonitrides of vanadium in the austenite.

The investigation results of the new steel hardness and the retained austenite volume fraction changes caused by the tempering temperature, obtained after the previous water quenching from a temperature of 1160°C are shown in Fig. 3. These assessments were done for the tempering temperature range: 100÷700°C.

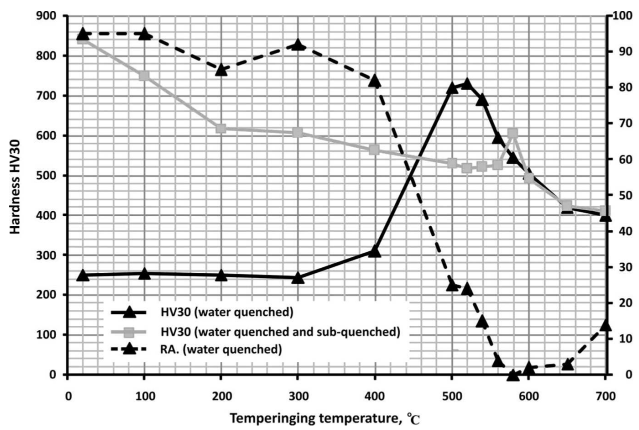
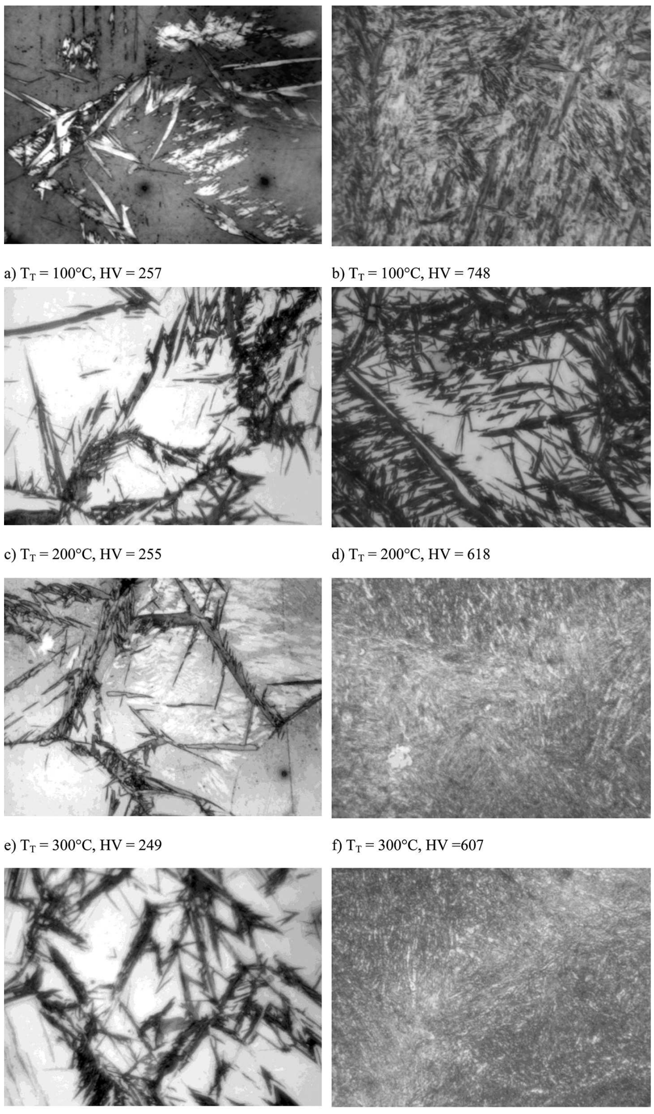


Fig. 3. Changes of the hardness and the retained austenite volume fraction caused by the tempering temperature after water quenching from a temperature of 1160°C of the new steel

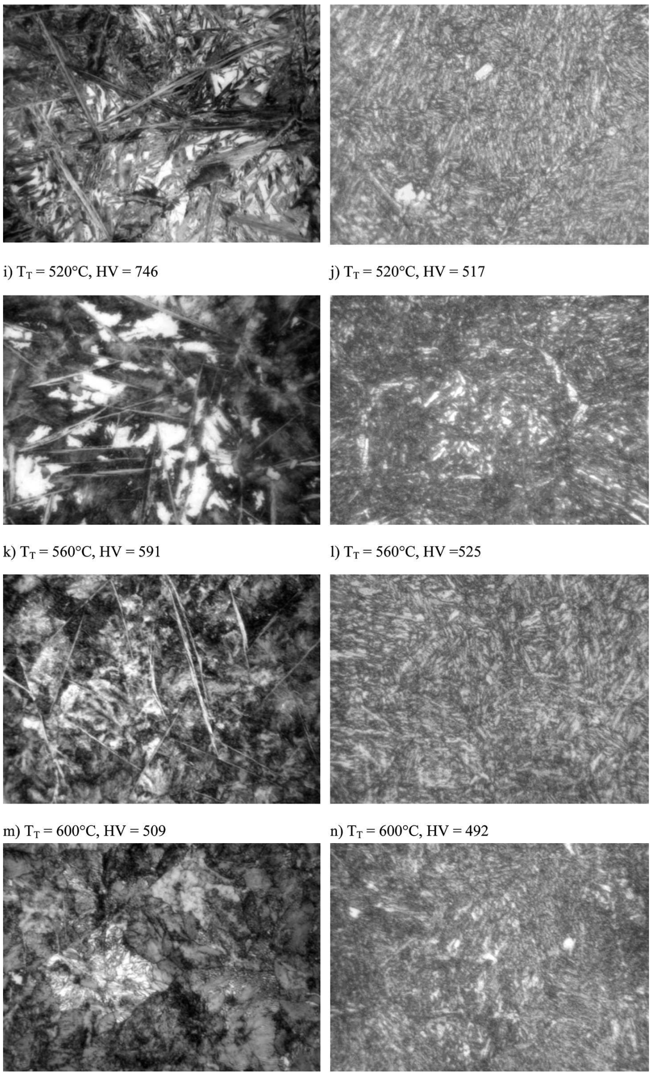
As can be seen (Fig. 3) the low hardness (257÷325 HV) of only water quenched samples containing in the solution (austenite) 1.25%C, 2.05%Mn, 1.61%Cr, 0.39%Mo and 0.44%V – in the tempering temperature range 100÷400°C – and the characteristic peak on the curve (already after exceeding a temperature of 300°C) are related to the presence (after quenching from a high austenitising temperature) of large amounts of the retained austenite followed by its transformation into the fresh martensite. An increased hardness above the tempering temperature of 400°C is also related, in a certain degree, to the secondary hardness caused by independently nucleating carbides of MC type. The hardness maximum (746 HV) related to the secondary hardness effect was detected at a tempering temperature of 520°C. Samples sub-quenched in liquid nitrogen, directly after the quenching treatment, indicated a decisively higher hardness (748÷563 HV) within the tempering temperature range to app. 400°C, which corresponds to the occurrence of a smaller volume fraction of the retained austenite in their structure. In one case (at tempering at 200°C) the hardness is slightly lower than could be expected and equals only 618 HV. This can be the result of a smaller transformation of the retained austenite (see Fig. 3).

The analysis of Figure 3 indicates that in samples water quenched from a temperature of 1160°C as well as in those which were tempered at 100°C (without sub-quenching), the retained austenite volume fraction equals app. 95%. The increased volume fraction of this austenite at a temperature of 700°C should be related to the possibility of a local exceeding of Ac_{1p} temperature (720°C).

The detailed metallographic documentation of the selected samples tempered within the temperature range: 100÷650°C, after the previous water quenching from a temperature of 1160°C, are presented in Figure 4 – for comparison - together with the samples tempered after sub-quenching in liquid nitrogen directly after the quenching treatment. Changes occurring in the steel microstructure confirm its hardness changes with the tempering temperature (see Fig. 3).



a) T_T = 100°C, HV = 257
b) T_T = 100°C, HV = 748
c) T_T = 200°C, HV = 255
d) T_T = 200°C, HV = 618
e) T_T = 300°C, HV = 249
f) T_T = 300°C, HV =607
g) T_T= 400°C, HV = 325
h) T_T = 400°C, HV = 563



i) T_T = 520°C, HV = 746 j) T_T = 520°C, HV = 517
k) T_T = 560°C, HV = 591 l) T_T = 560°C, HV =525
m) T_T = 600°C, HV = 509 n) T_T = 600°C, HV = 492
o) T_T = 650°C, HV = 426 p) T_T = 650°C, HV = 423

Fig. 4. Photographs of the microstructures of the tested alloy selected samples tempered in the temperature range: 100÷650°C, previously water quenched from a temperature of 1160°C, without sub-quenching (left side – Fig. a, c, e, g, i, k, m, o) and with sub-quenching (right side – Fig. b, d, f, h, j, l, n, p), magnification 630x, etching: nital 2%

5. Conclusions

1. The steel hardness increases with the austenitising temperature increase, achieving the maximum value of app. 895 HV (for only water quenched samples) and app. 937 HV (for sub-quenched samples) – for the austenitising temperature 800°C, and then – in both cases – starts decreasing. In case of only water quenched samples a very intensive hardness decrease should be related to the increase of the retained austenite volume fraction.
2. The retained austenite volume fraction determined by means of the X-ray method was equal app. 95% in samples quenched from the temperature of the total solubility of vanadium carbides and carbonitrides (MC type) in the austenite (1160°C). With an increase of the tempering temperature the volume fraction of this phase decreases, especially intensely after exceeding 400°C.
3. Quenching of the new Mn-Cr-Mo-V steel from the temperature of the total solubility of vanadium carbides and carbonitrides (MC type) in the austenite, causes a significant hardness increase during tempering (starting from a temperature of app. 400°C). This hardness increase corresponds to the transformation of large amounts of the retained austenite into the fresh martensite and – to a certain degree – to the secondary hardening by independently nucleating carbides, MC type.
4. The obtained investigation results will be used in future at designing the chemical composition and microstructures of the new hypereutectoid alloyed steels of properties required and expected by their users.

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Received: 20 September 2012.