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ARCHIVES OF METALLURGY AND MATERIALS

Volume 60 2015 Issue 1

DOI: 10.1515/amm-2015-0082

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THERMAL STABILITY OF NANOCRYSTALLINE STRUCTURE IN X37CrMoV5-l STEEL

STABILNOŚĆ CIEPLNA STRUKTURY NANOKRYSTALICZNEJ W STALI X37CrMoV5-1

The aim of the study was to investigate the thermal stability of the nanostructure produced in X37CrMoV5-1 tool steel by austempering heat treatment consisted of austenitization and isothermal quenching at the range of the bainitic transformation. The nanostructure was composed of bainitic ferrite plates of nanometric thickness separated by thin layers of retained austenite. It was revealed, that the annealing at the temperature higher than temperature of austempering led to formation of cementite precipitations. At the initial stage of annealing cementite precipitations occurred in the interfaces between ferritic bainite and austenite. With increasing temperature of annealing, the volume fraction and size of cementite precipitations also increased. Simultaneously fine spherical Fe_7C_3 carbides appeared. At the highest annealing temperature the large, spherical Fe_7C_3 carbides as well as cementite precipitates inside the ferrite grains were observed. Moreover the volume fraction of bainitic ferrite and of freshly formed martensite increased in steel as a result of retained austenite transformation during cooling down to room temperature.

Keywords: nanocrystalline structure, carbide free bainite, tool steel, thermal stability, TEM

Celem pracy było zbadanie stabilności cieplnej nanostruktury wytworzonej w stali narzędziowej X37CrMoV5-1 za pomocą obróbki cieplnej polegającej na austenityzacji i hartowaniu z przystankiem izotermicznym w zakresie przemiany bainitycznej. Utworzona nanostruktura składała się z płytek ferrytu bainitycznego nanometrycznej grubości rozdzielonych cienkimi warstwami austenitu szczątkowego. Ujawniono, że wyżarzanie stali w temperaturze wyższej niż temperatura przystanku izotermicznego prowadzi do wytworzenia w nanostrukturze wydzielenia cementytu. W początkowym etapie wyżarzania wydzielenia cementytu utworzyły się na granicach ferrytu bainitycznego i austenitu. Ze wzrostem temperatury wyżarzania następował wzrost udziału objętościowego i wielkości wydzieleń cementytu. Jednocześnie pojawiły się drobne wydzielenia węglika Fe_7C_3. Po wyżarzaniu w jeszcze wyższych temperaturach zaobserwowano duże, kuliste wydzielenia węglika Fe_7C_3 oraz wydzielenia cementytu w obrębie ziaren ferrytu. udział objętościowy tej fazy w stali. Nastąpił również wzrost udziału objętościowego ferrytu bainitycznego oraz świeżo utworzonego martenzytu w blokach w wyniku przemiany austenitu szczątkowego podczas chłodzenia stali do temperatury pokojowej.

1. Introduction

A new method of nanocrystalline structure formation in steels has been recently developed [1-4]. The method consists in a properly designed austempering heat treatment which allows obtaining steel of a nanobainitic structure with high strength and beneficial service properties [1-4, 7, 8] Such a material ensures a better compromise between plasticity and strength as compared to steel after a conventional quenching and tempering heat treatment. Until now a nanobainitic structure was obtained in special steel of properly designed chemical composition [5]. However, it has been shown recently that the production of the nanobainitic structure, so called nanobainitisation can improve strength and cracking resistance of some standard steel grades, e.g. X37CrMoV5-l hot work tool steel [7]. The nanocrystalline structure similarly to martensite is a thermodynamically metastable, thus it is important to know its thermal stability [9]. This study aims at determining whether and to what extent the structure of nanocrystalline bainite is stable at increased temperatures and what microstructural changes occur. In order to achieve this objective the dilatometric investigations were carried out together with the microstructural observations by transmission electron microscopy (TEM). For the phases identification and description the bright and dark field techniques combined with diffraction pattern analysis were used.

2. Experimental procedure

X37CrMoV5-l hot-work tool steel of the following chemical composition (in weight %): C-0.37, Si-1.01, Mn-0.38, Cr-4.91, Mo-1.2, V-0.34, Ni-0.19 was subjected to austempering at 300°C. This heat treatment produced a structure of carbide-free bainite of nanometric plates width separated by layers of retained austenite. In order to determine ther-

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mal stability of the nanostructure, the steel was annealed in a dilatometer under protective atmosphere at 400, 500 and 600°C for 8 hours. During the annealing the changes in sample length as a function of time and temperature were recorded. All heat treated samples were observed by means of transmission electron microscope using bright field (BF) and dark field (DF) imaging combined with the analysis of diffraction patterns. In order to determine volume fraction and grain size of particular phases the methods of stereological analysis were used.

The thickness of ferrite plates and austenite layers visible on the TEM images was determined using the following formula:

$$d = \frac{2}{\Pi} L \tag{1}$$

where d is actual dimension of the microstructure element, L – dimension of this element measured on TEM image [6].

Volume fraction of the phases were calculated with the assumption that the volume content of a given phase is equivalent to its section area in the microstructure image plane. A number of secant lines of the length l was applied to images of microstructure intersecting with specific phase n-times. The following formula was used to calculate volume fraction of selected phases:

$$\mathrm{V_v} = \frac{\Sigma \mathrm{c_{ik}}}{\mathrm{l}} \tag{2}$$

where $\sum c_{ik}$ is the sum of the widths of all intersections of the secant line l with a given phase, l – length of the secant line.

The microstructural observations were analysed and correlated with the dilatometric curves obtained during the annealing of steel samples.

3. Results and discussion

Isothermal quenching of X37CrMoV5-l steel at 300°C allowed to produce a structure of carbide-free bainite composed of bainitic ferrite plates separated by the layers of retained austenite (Fig. 1). The average thickness of bainitic ferrite plates is 89 nm±6 nm and the average thickness of residual austenite layers is of 31 nm±2 nm. The examples of the microstructure are shown in Fig. 1 and in Fig. 2. The volume fraction of ferrite is 57% and the rest being austenite both in the form of layers and blocks (Fig. 2). The cross section area of the austenite blocks varies from 0.13 $\mu\mathrm{m}^2$ to 4.33 $\mu\mathrm{m}^2$. The austenite blocks are partially transformed to fresh martensite.

The annealing of austempered samples in a dilatometer resulted in various dilatational changes (Figs. 3, 4). It was observed that with increasing the temperature of the annealing process, the changes on the dillatometric curves are greater, indicating greater changes in the microstructure. The analysis of dilatograms of steel annealed at 400°C showed only small dilatational changes occuring for about 1 minute, immediately after reaching the set temperature. It means that only minimal changes occurred in the microstructure of the annealed sample [10]. Increase of temperature to 500°C led to a significant shrinkage of the sample, most probably related to the precipitation of carbides [11].

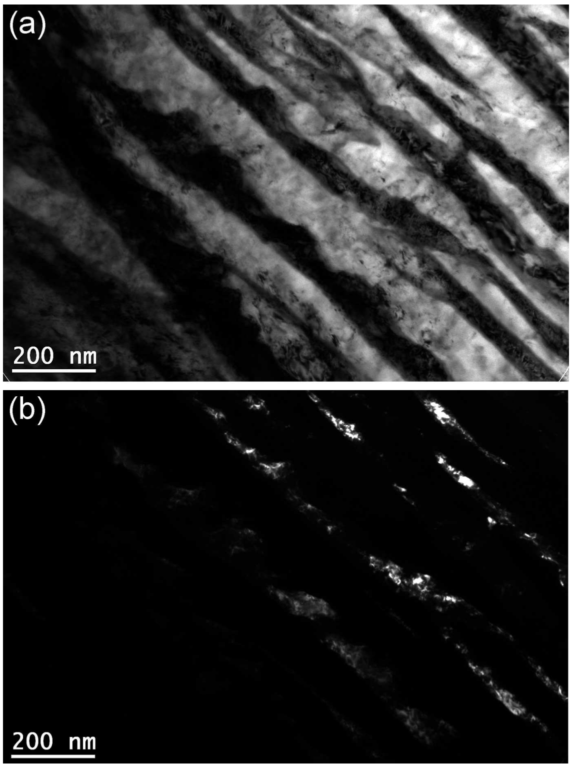

Fig. 1. Microstructure of X37CrMoV5-1 steel after austmpering at 300°C, (b) – dark field image of austenite

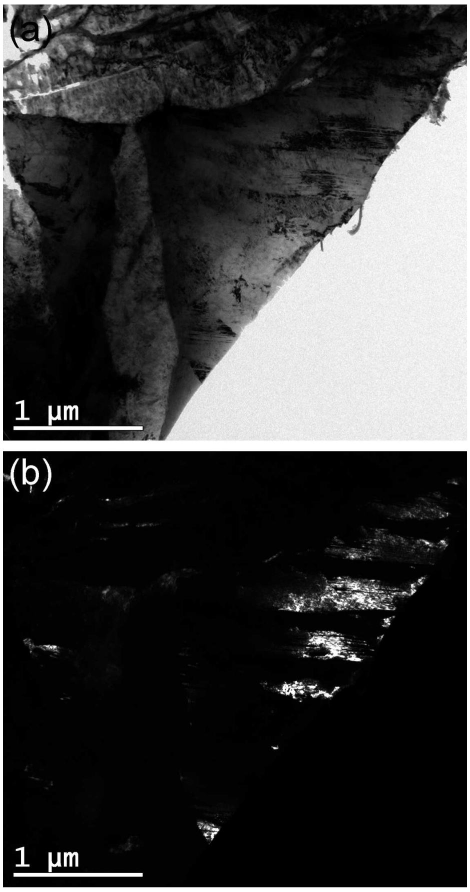

Fig. 2. (a) the block of retained austenite with partial martensitic transition in X37CrMoV5-1 after austempering at 300°C, (b) – dark field image of ferrite

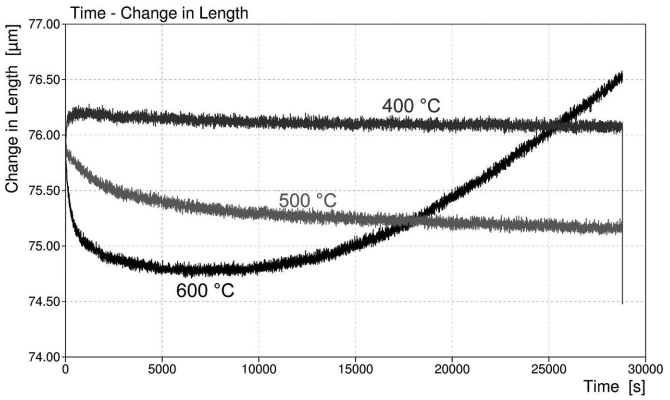

Fig. 3. Dilatometric curves presenting a comparison of sample length changes during 8-hour isothermal annealing at various temperatures

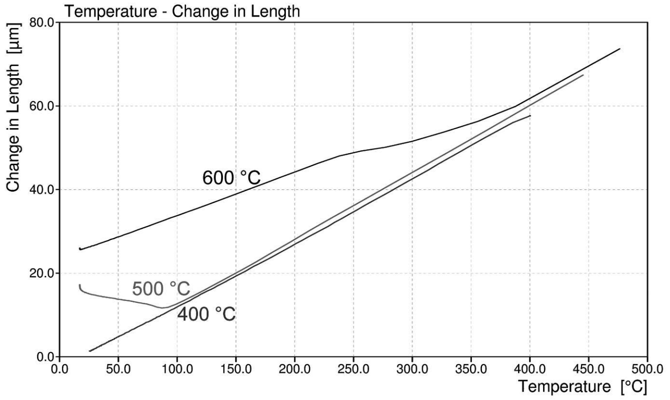

Fig. 4. Dilatometric curve recorded during cooling after 8-hour isothermal annealing at various temperatures

The annealing at 600°C led to a shrinkage twice as high as at 500°C during the initial stage of heat treatment. This effect was finished after 2 hours of heat treatment and subsequently elongation of the sample started to occur. The observed increase in length of the sample could be caused by austenite transformation [11], or, which is more likely, by the formation of another type of carbides nucleating independently. Also a dilatational effect related to retained austenite transformation to fresh martensite in case of heat treatment at 500°C was observed during the cooling of the samples to room temperature. In the case of the annealing at 600°C a bainitic transformation were recorded during final cooling of the sample to room temperature (Fig. 4). The above findings confirm that in X37CrMoV5-l steel with increasing temperature of annealing, the destabilisation of austenite occurs. This effect is due to the decrease of carbon content in the austenite caused by the carbides precipitation.

The transmission electron microscopy observations are consistent with the dilatometric examination results. Annealing at the temperature of 400°C only slightly affects the microstructure. The typical nanobainitic areas, composed of ferrite plates with the average width of 116 nm±5 nm separated by austenite layers with an average thickness of 32 nm±2 nm (Figs. 5, 6) were still observed. The size of the austenite blocks, partially transformed to fresh martensite, reached the area up to 5 μm^2 (Fig. 8a). Reflections of cementite on the diffraction patterns were also observed. The cementite precipitates occurred mainly on the ferrite/austenite interfaces (Fig. 6).

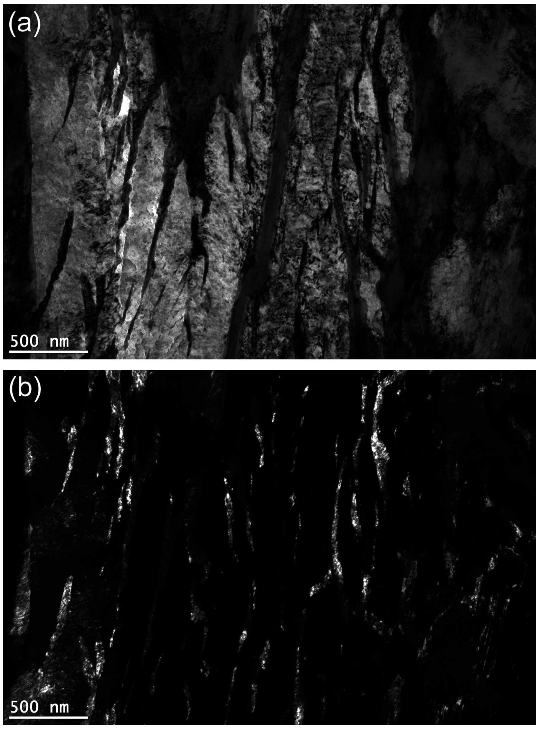

Fig. 5. Microstructure of X37CrMoV5-1 steel after austempering at 300°C and annealing at 400°C, (b) – dark field image of austenite

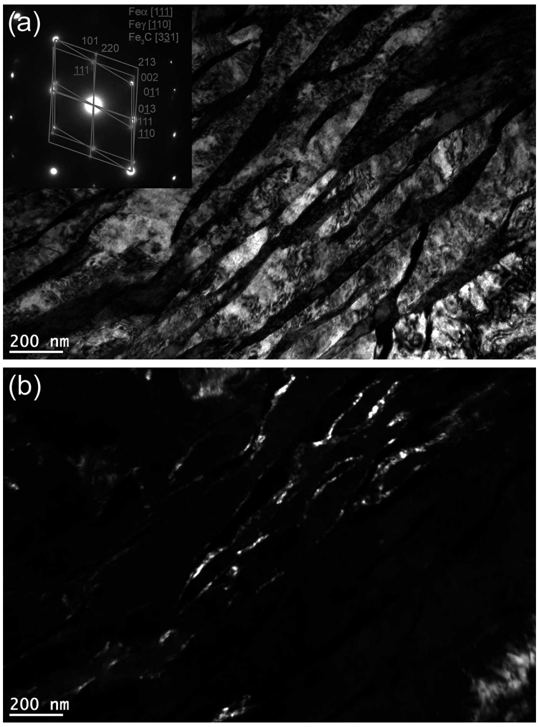

Fig. 6. Microstructure of X37CrMoV5-1 steel after austempering at 300°C and annealing at 400°C, (b) – dark field image of cementite

The increase of the annealing temperature to 500°C caused a reduction in the observed nanobainitic areas, and a slight growth of the bainitic ferrite laths, up to 126 nm±10 nm. On the contrary the width of the austenite remained almost constant during annealing. Their thickness is 33 nm±5 nm, which is approximately equal to the austenite layers after initial austempering and after annealing at 400°C. At the same time the volume fraction of the bainitic ferrite slightly increased from 62.7±3.9 % after annealing at 400°C to 64±5.5 % to after annealing at 500°C. The rest is the retained austenite in the form of layers and blocks partially transformed into fresh martensite (Fig. 8b). The cementite precipitates on the ferrite/austenite interface were still observed, as well as the rare, spherical Fe_7C_3 carbides. The microstructure of steel after annealing at 500°C is presented on the Figure 7.

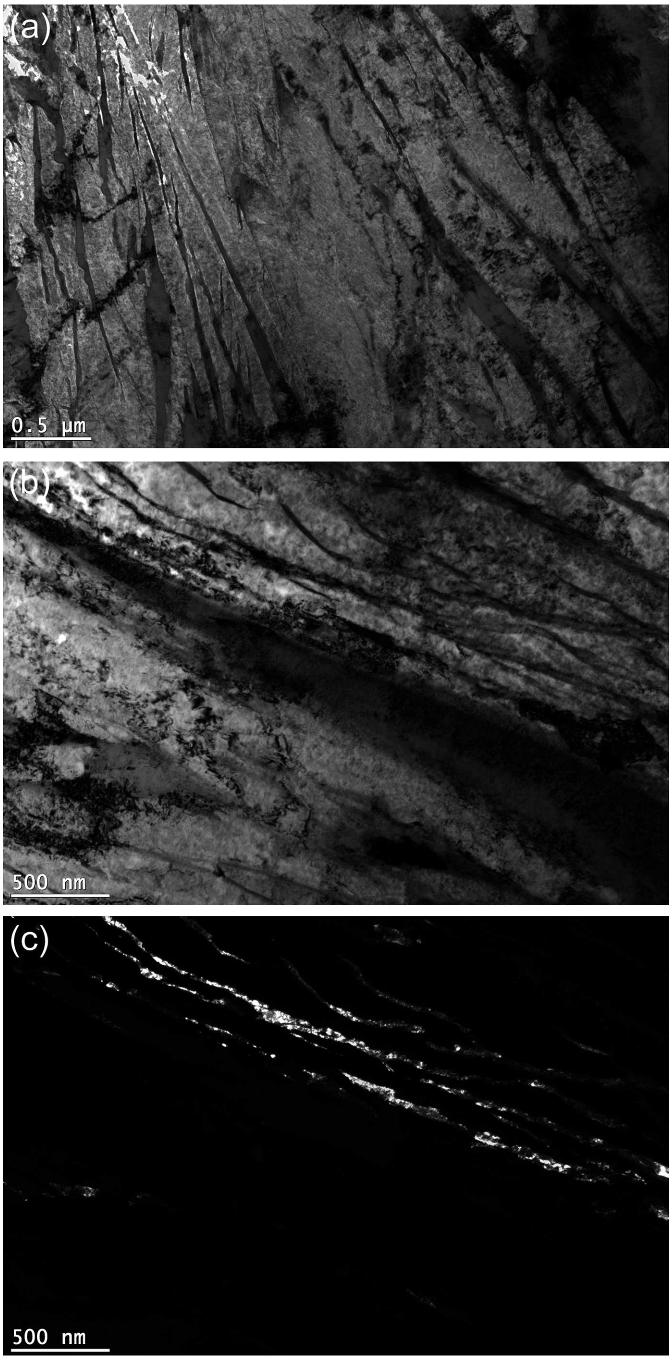

Fig. 7. Microstructure of X37CrMoV5-1 steel after austempering at 300°C and annealing at 500°C – (a), (b), dark field image of austenite (c)

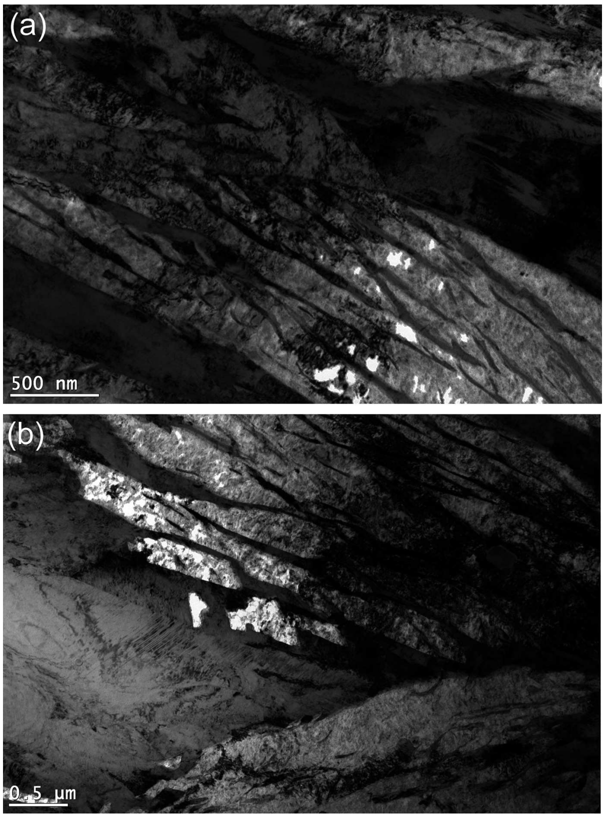

Fig. 8. The block of retained austenite and partial fresh martensite in X37CrMoV5-1 after austempering at 300°C and annealing at: 400°C (a), 500°C (b)

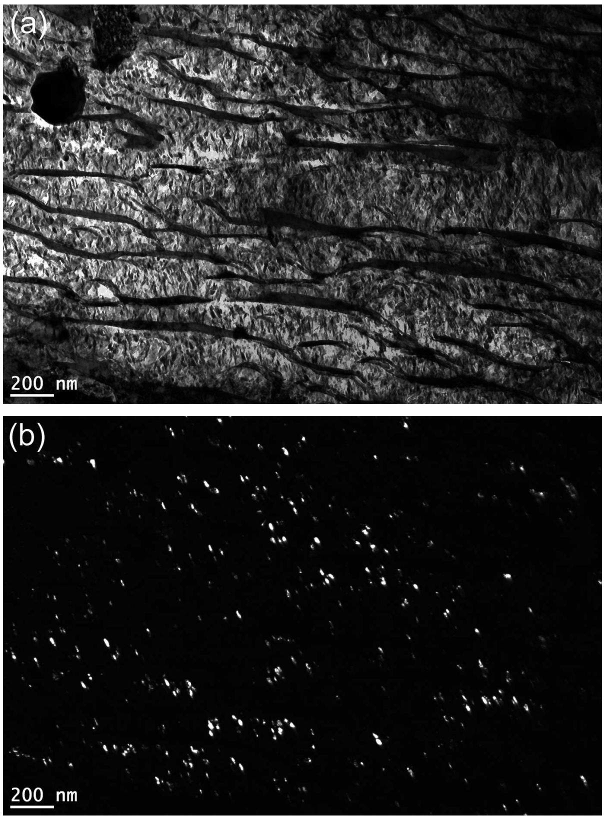

Fig. 9. Microstructure of X37CrMoV5-1 steel after austempering at 300°C and annealing at 600°C, (b) – dark field image of cementite

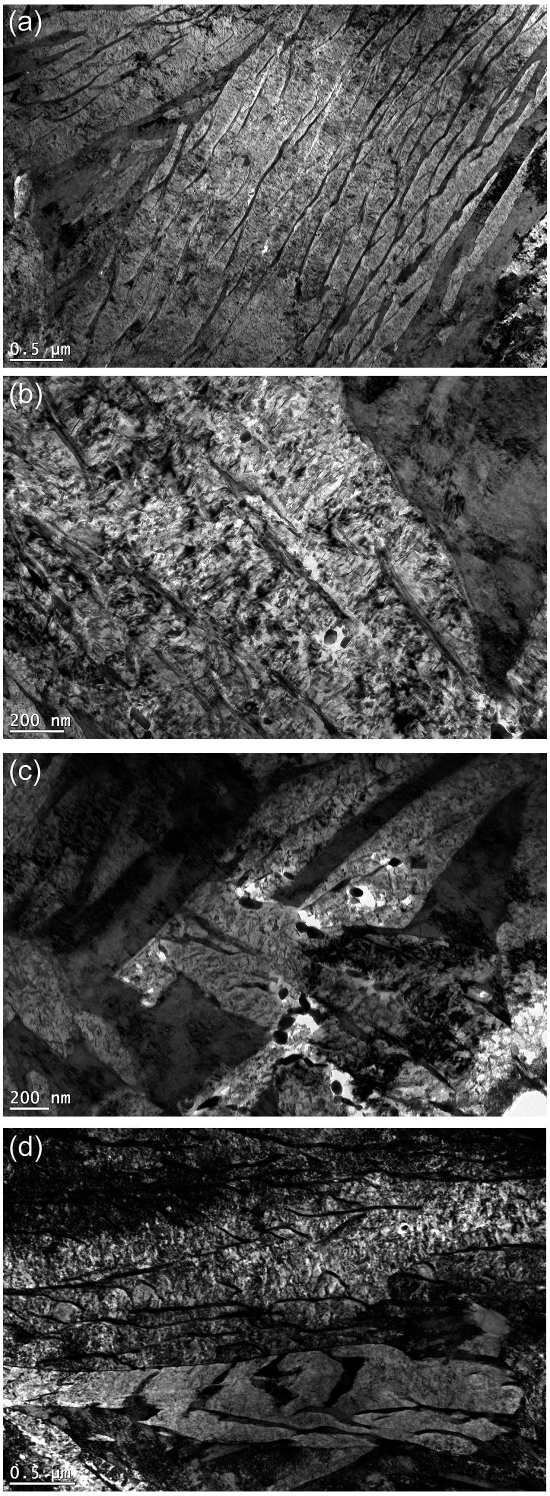

Fig. 10. Microstructure of X37CrMoV5-1 steel after austempering at 300°C and annealing at 600°C (a-d)

Further increase of the annealing temperature up to 600°C led only to a small increase of width of the retained austenite layers (the average thickness of the austenite layers is 41 nm±3 nm) but caused further growth of ferrite plates width up to 160 nm±10 nm in average as well as the reduction of initial nanobainitic structure (Figs. 9-11). The ferrite content increased up to 73.6%±3.8%. Fine dispersed precipitates of cementite occur inside the ferrite laths (Fig. 9). The density of observed spherical Fe_7C_3 carbides is higher as compared to their density in steel after annealing at 500°C.

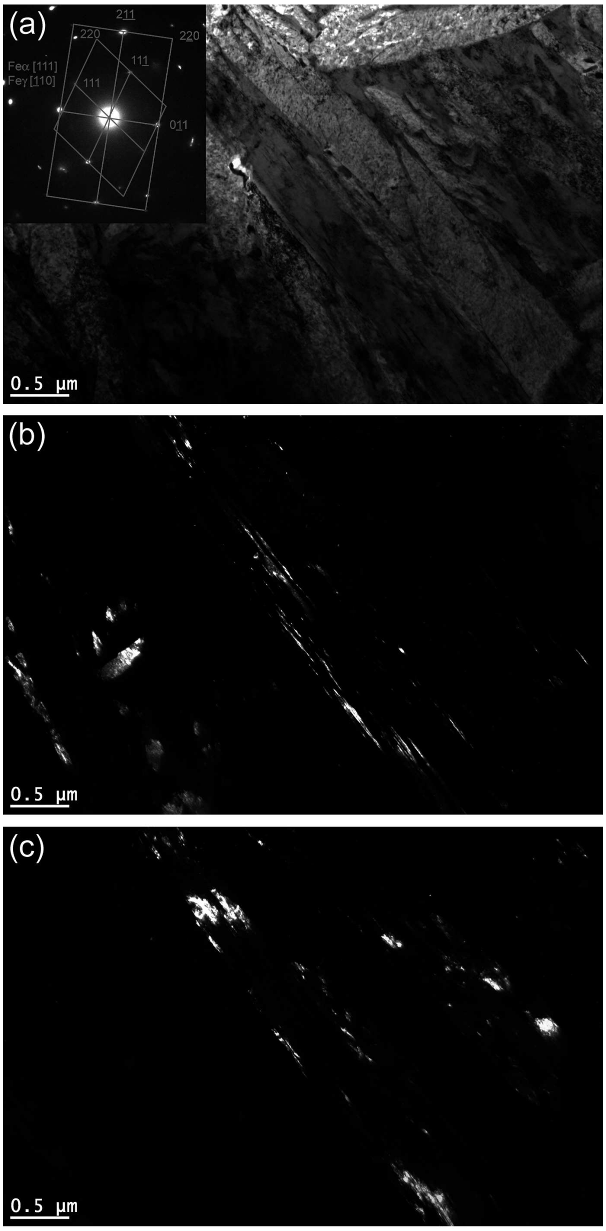

Fig. 11. The block of austenite and fresh martensite in X37CrMoV5-1 after austempering at 300°C and annealing at 600°C (a) – BF, (b) – DF of austenite, (c) – DF of ferrite

It can be concluded that annealing at the temperature range from 400°C to 600°C leads to precipitation of carbides, mainly cementite. Very fine carbide precipitates form at lower annealing temperatures. Their volume fraction and size increase with the annealing temperature. In the material annealed at 600°C large, spherical precipitates of Fe_7C_3 carbide and finely-dispersed Fe_3C carbides inside the ferrite grains were observed. The carbide precipitation leads to a decrease in the carbon content of austenite and consequently in a reduction of the austenite stability. Therefore, during cooling down to room temperature the austenite transforms to fresh martensite or bainite. As a result of the phase transformation of austenite there are less and less regions of a nanobainitic structure (fine ferrite plates separated by retained austenite layers) and the content of ferrite in steel as well as the size of ferrite grains increase with the increasing temperature of annealing.

4. Conclusions

The microstructure investigation and the dilatometric results examination revealed that annealing at temperatures above 400°C deteriorates the nanobainitic structure in X37CrMoV5-l steel. Therefore steels containing a nanobainitic structure cannot be used for high temperature applications. However, this steel can be used for manufacturing elements working at temperatures below 400°C.

It was found that the changes occurring in nanobainitic structure during annealing are due to the carbides precipitation. This process led to a decrease of carbon content in retained austenite and decrease its stability which results in the transformation into bainite or fresh martensite during cooling down to room temperature.

Acknowledgements

The results presented in this paper have been obtained within the project "Production of nanocrystalline steels using phase transformations" – NANOSTAL (contract no. POIG 01.01.02-14-100/09 with the Polish Ministry of Science and Higher Education). The project is co-financed by the European Union from the European Regional Development Fund within Operational Programme Innovative Economy 2007-2013.

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Received: 20 March 2014.