TEMPERATURE STABILITY OF CARBIDE-FREE NANOBAINITE WITH RESIDUAL AUSTENITE IN X37CrMoV5-1 STEEL

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Abstract

The subject of this study is the temperature stability of a microstructure in X37CrMoV5-1 hot-work tool steel. Conventionally treated by quenching and high-tempering this steel is used for tools working at temperature up to 550 °C. In this study a non-conventional treatment consisting of austempering at 300 °C, which is slightly higher than martensite start temperature Ms = 293 °C, was used. As a result a microstructure of carbide-free nanocrystalline bainite with residual austenite was created. This microstructure ensures a very good compromise between high strength and plasticity at room temperature. The aim of this study was to determine temperature stability of the obtained microstructure in a range of temperatures 350 - 600 °C, which are higher than the temperature of the austempering treatment. For this purpose steel samples after austempering were subjected to dilatometric tests during continuous heating and isothermal annealing at various temperatures. Phase transformations were recorded during cooling of the samples after continuous and isothermal heating. These investigations allowed us to determine the highest temperature up to which any phase transformation was not observed to occur during dilatometric tests. The changes occurring in the carbide-free nanobainite with residual austenite after exceeding the critical temperature were characterised. The impact of the observed changes occurring in the microstructure above the critical temperature on the mechanical properties of steel was discussed.

Keywords: Dilatometric test; Phase transformations; Heat treatment; Carbide-free bainite; Nanocrystalline structure; Thermal stability

1. INTRODUCTION

A treatment of quenching, with an isothermal stop in the temperature range of the bainitic transformation allows to create a nanocrystalline bainite (nanobainite) with residual austenite in X37CrMoV5-1 hot-work steel [1]. The nanobainitic structure ensures the steel's beneficial mechanical properties at room temperature. Since this steel is most often used for tools working at increased temperatures, it is critical to determine temperature stability of the nanobainite. The effect of temperature on nanobainite stability was characterised e.g. in references [2], [4], [5], [6]. It was shown that the rise of temperature causes residual austenite to decay into ferrite and cementite. Depending on the studied steel's chemical composition, the temperature, at which the austenite lost its stability was at least 350 °C and in most cases over 400 °C. However, the published papers concern steels of a chemical composition significantly different from the studied X37CrMoV5-1 steel's composition. In order to assess the potential of this steel for hot-work use after its nanostructuring treatment, a range of temperatures was determined in this study, in which the carbide-free nanobainite created during isothermal quenching in X37CrMoV5-1 steel remains stable. The type of transformations occurring during annealing at increased temperatures was also determined. The structural changes observed in X37CrMoV5-1 steel were compared with those characterised in references and occurring in other nanobainitic steels.

2. MATERIALS AND METHODS

The samples of X37CrMoV5-1 steel have the following chemical composition (wt %): 0.37C, 1.01Si, 0.38Mn, 4.91Cr, 1.20Mo, 0.34V, 0.19Ni. Steel was submitted to a thermal treatment consisting on austenitizing at 1030 °C followed by quenching down to 300 °C and isothermal annealing at this temperature for 19 hours. As a result a mixture of carbide-free bainite and untransformed austenite was created in the studied steel. The volume fraction of bainite was 60 %, and the remaining 40% was austenite partially transformed into martensite. The austenite occured both as thin layers placed between plates of bainitic ferrite and in blocks. The chemical composition rendered impossible the formation of carbides during the isothermal annealing. This favoured the diffusion of, carbon during bainitic transformation from the regions of newly formed ferrite into austenite, which ensured partial stability of the austenitic phase after cooling to ambient temperature.

A nanobainitic structure is characterised by high values of strength parameters, ensuring a beneficial compromise between tensile strength and hardness on one hand and ductility, resilience and cracking resistance on the other [7]. Moreover this type of structure shows high stability at increased temperature [6], [2], [5]. Since the studied steel is commonly used as hot-work steel, its stability at increased temperatures should be very high.

All dilatometric studies were conducted with a Baehr DIL805L quenching dilatometer. The device uses a conductor for heating and a jet of compressed helium for cooling. The studied samples were shaped as 10 mm long cylinders of 3 mm diameter. Temperature stability tests were conducted during continuous heating at 0.3 °C/min as well as during isothermal annealing at 300 °C, 400 °C, 500 °C and 600 °C. The isothermal annealing lasted for 8 hours.

The samples designed for continuous heating tests were preliminarily subjected to a heat treatment, which created the nanobainitic structure inside the dilatometer furnace in a single process combined with the thermal stability test. The samples designed for isothermal tests were cut out from steel that was subjected to isothermal quenching at 300 °C in a tin bath.

All the dilatometric tests were carried out in the helium atmosphere. The heating rate during the isothermal tests was 200 °C/s. After the annealing was over, the samples cooled down naturally, without the blow of gas or heating, but with the changes in their length were recorded all the time.

After the dilatometric tests, the hardness of the samples was measured using the Vickers method with a 2 kg load.

3. RESULTS OF DILATOMETRIC TESTS AND THEIR ANALYSIS

3.1 Continuous heating

The plot of length change of heat-treated steel during its heating as a function of temperature was shown in **Fig. 1**. The heating was carried out at a steady rate of 0.3 °C/min from ambient temperature to 700 °C. The differential curve was added to the plot for a more distinct illustration of the transformations occurred.

The differential curve shows very slight dilatational effects in a wide range of temperatures. Only at 620 °C a slight contraction caused probably by precipitation of M_3C carbide was observed. At 650 °C sample elongation caused by transformation of austenite contained in the sample began. This process stopped above 700 °C, which was not recorded in the dilatogram.

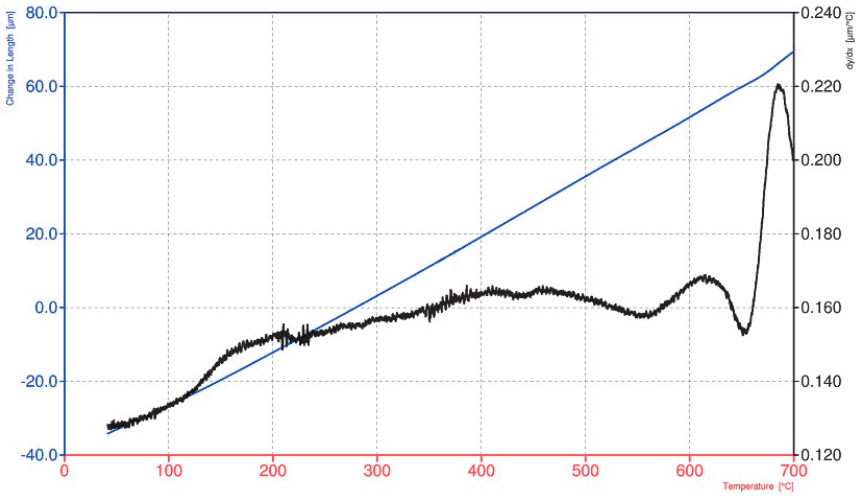

Fig. 1 Dilatometric dependence with differential curve presenting transformations that occur during continuous heating

Both mentioned processes (carbide precipitation and austenite decay) probably overlap, due to which the quoted temperature of the start of austenite transformation might be slightly inaccurate.

3.2 Isothermal annealing

Since no distinct dilatational effects occurred at low temperatures, the isothermal tests were carried out at the temperatures higher than the temperature of heat treatment. The dilatograms derived from 8-hour annealing at 400, 450, 500 and 600 °C are shown in **Fig. 2**.

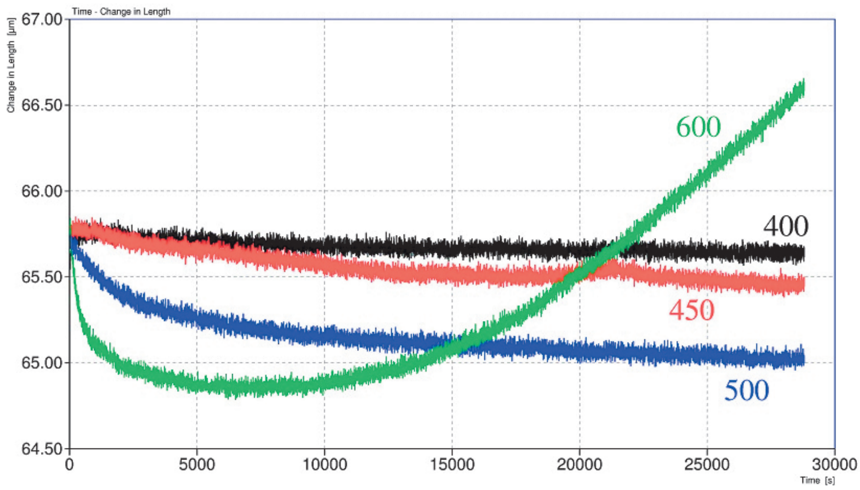

Fig. 2 Dilatometric plot presenting a comparison of sample length changes during 8-hour isothermal annealing at 400, 450, 500 and 600 °C

In case of dilatometric curve obtained at 400 °C, the dilatational effect was at the threshold of measurement accuracy, which indicated either minimal changes in the structure or lack thereof. A minimal contraction observed at 450 °C could be related to slow precipitation of ε-carbides or cementite.

The increase of the temperature up to 500 °C led to a significant contraction caused by precipitation of carbides. This contraction proceeded slower and slower, and it was not more observed at the 8th hour of the test.

The sample annealed at 600 °C behaved differently. After 2 hours its contraction was twice as at 500 °C. Afterwards, it stopped, and a positive dilatational effect was observed, which resulted in the sample being longer after 8 hours of annealing than in its initial state. Elongation after 2 hours of annealing is connected with a change in the type of forming carbides, e.g. precipitation of independently nucleating MC or M_2C [3].

A very high rate of heating the sample up to the temperature of the isothermal stop prevents the accurate detection of dilatational changes. To reveal such changes a heating of 50 °C/s was carried out to the temperature of 400 °C. It revealed that the changes of sample length of about 0.1 μm presented in **Fig. 2** do not render the whole course of the process adequately. **Fig. 3** shows a magnified fragment of the dilatometric dependence plot the beginning of the isothermal annealing process.

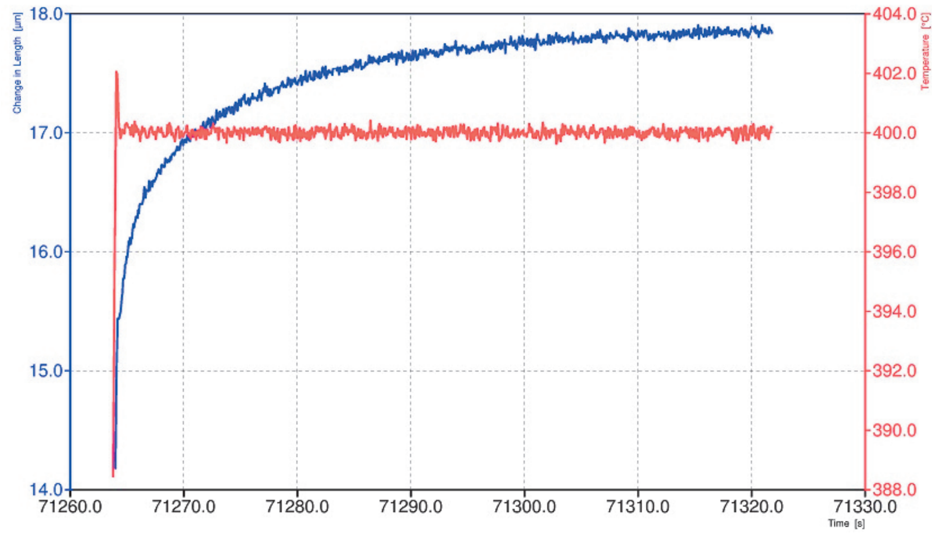

Fig. 3 Magnified fragment of the dilatometric plot representing the beginning of the isothermal annealing process at 400 °C

It appeared that directly after the sample reached the set temperature, its length rose by 2.5 μm during the first 50 seconds. Afterwards, the sample length stabilised and did not alter significantly for the remaining 8 hours. Also, no transformations were observed to occur before reaching the isothermal stop.

3.3 Cooling after isothermal annealing

After the annealing was over, the samples were cooled down to ambient temperature.

The samples annealed at lower temperatures, in which the dilatational effects were minimal, did not exhibit any phase transformations during cooling (**Fig. 4**). On the contrary, annealing at 500 and 600 °C resulted in destabilisation of the austenite contained in the tested samples. It was confirmed by a strong positive dilatational effect indicating that some phase transformations occurred during cooling.

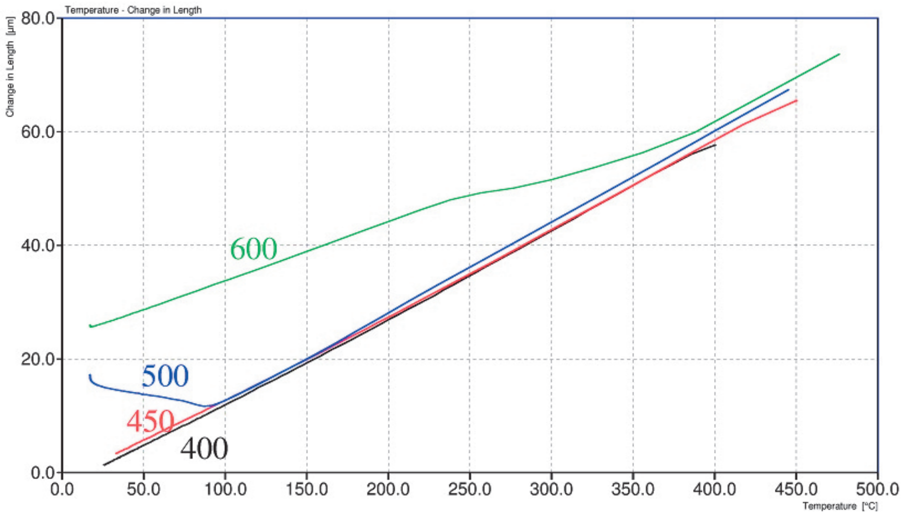

Fig. 4 Dilatometric plot recorded during sample cooling after 8-hour isothermal annealing at 400, 450, 500 and 600 °C

After annealing at 500 °C, a martensitic transformation began at about 90 °C, and came to an end below room temperature.

Annealing at 600 °C resulted in a bainitic transformation occurring during cooling. The transformation began at 385 °C and ended at 230 °C. Below no further phase transformations occurred till room temperature.

Since in the sample annealed at 600 °C the transformation of austenite during cooling began at a higher temperature than in the one annealed at 500 °C, and the final length of the sample was greater, it can be assumed that the higher temperature caused a greater destabilisation of residual austenite.

During the formation of carbide-free bainite, austenite was stabilized due to its enrichment with carbon derived from the forming ferrite, while the amount of other elements remained at a steady level [4]. Therefore, it can be concluded that the reduced stability of austenite during annealing resulted from a decrease in carbon content due to the, carbides formation at high temperatures of isothermal annealing, as it was shown in **Fig. 2**.

3.4 Hardness measurements

After the dilatometric tests, the samples were subjected to hardness measurements. The effect of the temperature of 8-hour isothermal annealing on hardness of the studied steel is shown in **Fig. 5**.

The hardness of a sample subjected to preliminary treatment of isothermal quenching with a structure of carbide-free bainite created was set as reference. Its value was 507 HV. Annealing in 400 °C did not significantly alter this value. Also annealing at 450 °C only slightly affects the hardness, which in this case reached 521 HV. Since during annealing and cooling no positive dilatational effects were observed, the hardness change was probably caused by formation of fine-dispersed carbide precipitations in the initial phase of isothermal annealing rather than by decrease in the content of soft austenitic phase.

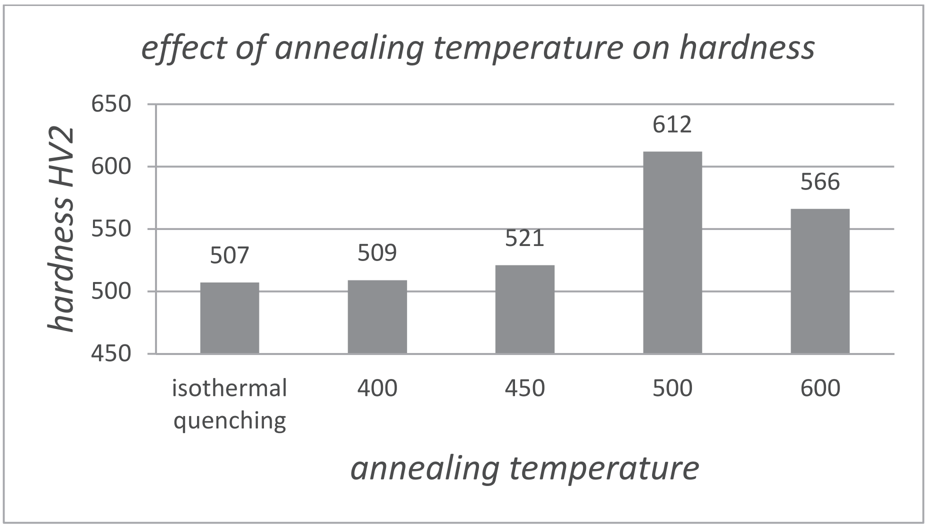

Fig. 5 Sample hardness relative to the temperature of 8-hour isothermal annealing

A substantial increase in hardness was observed after the annealing temperature had risen to 500 °C. The value of 610 HV mainly resulted from the martensitic transformation which occured during cooling of the material (as shown in **Fig. 4**). The strengthening related to carbide precipitation probably also occurred, yet its extent is impossible to determine. Annealing at 600 °C caused a lesser increase in the hardness, which reached 565 HV. This can be explained by a bainitic transformation that took place during cooling rather than a martensitic one, as it had been with annealing at 500 °C. Hardness can also be affected by coagulation of carbides, and hence decrease in their dispersion, which in turn lessens the strengthening. The change of the type of forming carbides is attested by different dilatational effects presented in **Fig. 2**.

CONCLUSIONS

The structure of X37CrMoV5-1 steel subjected to isothermal quenching is stable at up to 400 °C. Eight-hour annealing at 400 °C did not affect the hardness of the studied steel and caused small dilatometric effects only in its initial phase.

With annealing at 400 °C the phenomenon of secondary hardness related to precipitation of carbides from carbon-supersaturated austenite was observed.

At temperatures higher than 500 °C a decrease in the stability of residual austenite present in the steel was observed. This led to a martensitic transformation during cooling to room temperature. The martensite formed significantly increases the steel's hardness but can have a negative influence on its plasticity and cracking resistance.

The observed effects of residual austenite destabilisation and of secondary hardness, after they have been thoroughly studied, could find a use in modelling phase composition and properties of steels with a structure of carbide-free nanobainite.

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