THE KINETICS OF PHASE TRANSFORMATIONS DURING CONTINUOUS HEATING FROM AS-QUENCHED STATE OF 17-4PH STEEL

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Abstract

This work contains a detailed description of the influence of continuous heating from as-quenched state on microstructure development in the 17-4PH steel. The temperature ranges of the phase transformations occurring during heating from the as-quenched state (tempering) were determined from dilatometric tests. An interpretation of heating dilatometric curves was also presented. The temperature ranges of the occurred transformations during tempering of investigated steel were drawn on CHT (Continuous Heating Transformations) diagram. Moreover, the microstructure development in investigated samples, reflecting the extension of the phase transformations during tempering, was discussed as well. In as-quenched state the microstructure of investigated steel consist of low carbon martensite, ferrite δ banding, and niobium secondary carbides of MC type, which have not been dissolved during austenitizing. Changes in the microstructure of the investigated steel, during the heating from the as-quenched state result in precipitation of transition iron carbides, transformation of small amount of retained austenite into martensite, and precipitation of the Cu_{ε} phase. There were no significant changes in the chemical composition as well as in the morphology of the δ-ferrite.

Keywords: 17-4PH steel, tempering, kinetics, phase transformations, CHT diagram

1. INTRODUCTION

The study of phase transformations in alloys, a specially steels, is essential of primary importance for the optimization of thermal and thermomechanical treatments. One of the most popular techniques is dilatometry. A simple principle which consists of heating and/or cooling a material while measuring dimensional changes as a function of temperature or time has been studied. Dilatometry allows monitoring of any bulk phase transformation occurring in metals and alloys, such transformations being detected as soon as the densities of phases differ [1-8]. Dilatometry is commonly used for intermetallic phase precipitation kinetics analysis of precipitation hardening alloys, such as nickel based alloys [8-11].

Precipitation-hardened stainless steels were first developed during the 1940s, and since then, they have become increasingly important in a variety of applications in which their special properties can be utilized. The most important of these properties are ease of fabrication, high strength, relatively good ductility, and excellent corrosion resistance. The 17-4 PH (precipitation hardening) stainless steel is a martensitic stainless steel containing up to 5 wt. % Cu and is strengthened by the precipitation of highly dispersed copper particles in the low carbon martensite matrix. This steel is more common than any other type of precipitation hardened stainless steels and thus has been used for variety of applications including oil field valve parts, chemical process equipment, aircraft fittings, pump shafts, gears, paper mill equipment, and jet engine parts [12-15].

2. EXPERIMENTAL

The research was performed on 17-4PH steel. The chemical composition of investigated steel is given in **Table 1**. The dilatometric investigations were performed using the Adamel DT1000 high-speed dilatometer. Specimens of 2 mm in diameter and height of 12 mm, were austenitized for 20 minutes at 1040 °C, quenched and heated to 700°C at a rate ranging between 0.05 and 35 °C/s. Additionally, the quenched samples were heated with a rate of 0.05 °C/s up to chosen temperatures. The microstructure of the investigated steel was examined by a light microscope Axiovert 200 MAT and the Zeiss scanning electron microscope with Tident XM 4 system of the EDAX Company - energy dispersive X-RAY (EDS) analysis and JEM 200CX transmission electron microscope.

Table 1 Chemical composition of the investigated steel (wt. %)

Grade	C	Mn	Si	P	S	Cr	Ni	Nb	Cu	Fe
17-4PH	0.04	0.88	0.32	0.022	0.002	15.96	4.31	0.26	3.53	bal.

The microstructure of the investigated steel in as-delivered condition is presented in **Fig. 1**. The microstructure consist of tempered martensite, ferrite δ banding (**Fig. 1b**) as well as niobium secondary carbides of MC type. Hardenss of the steel in as-delivered condition is 366 HV10. Microstructure of investigated steel after quenching from 1040 °C is presented in **Fig. 2**. In as-quenched state the microstructure of the investigated steel consist of low carbon martensite, ferrite δ banding (**Fig. 2b**) as well as niobium secondary carbides of MC type which have not been dissolved during austenitizing (**Fig. 2c**). Hardness after quenching is close to the hardness in as-delivered condition, and equal 360 HV10.

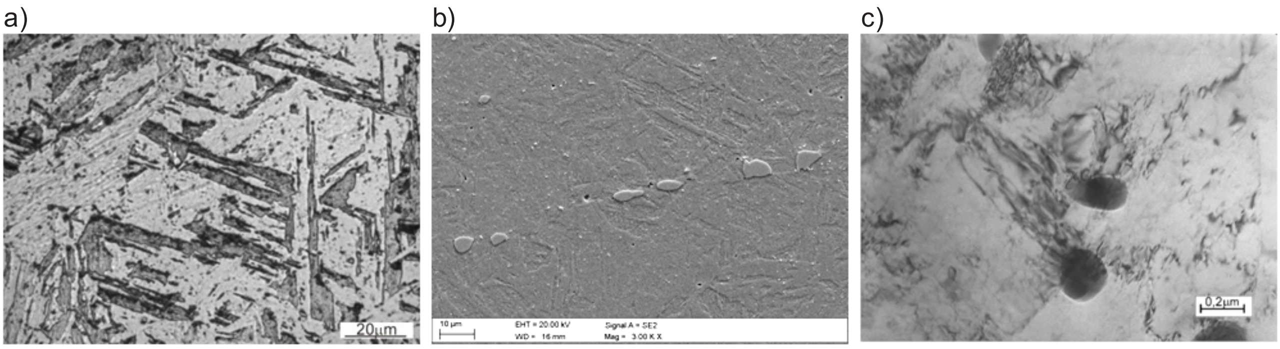

Fig. 1 Microstructures of the 17-4PH steel in as-delivered condition: a) Light microscope, b) SEM, c) TEM

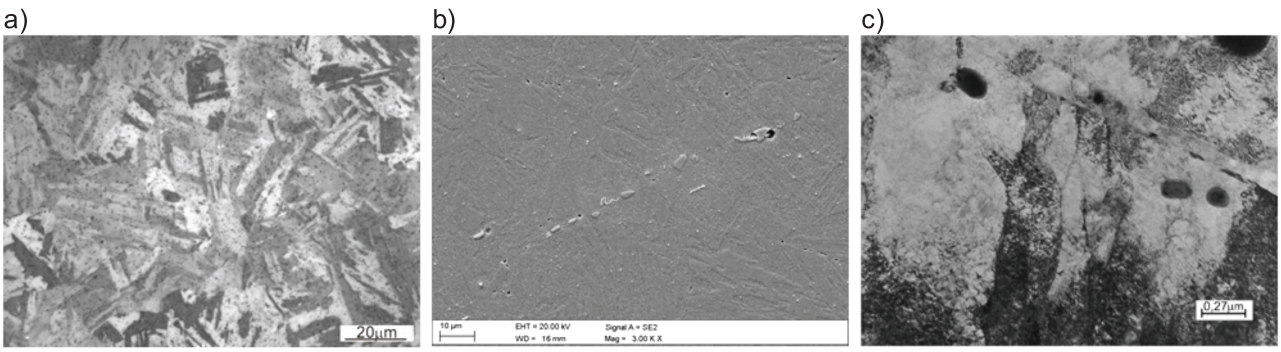

Fig. 2 Microstructures of the 17-4PH steel after quenching from 1040 °C: a) Light microscope, b) SEM, c) TEM

3. RESULTS AND DISSCUSION

Fig. 3 shows a dilatogram of the quenched samples heated with a rate of 0.5 °C/s, along with the corresponding differential curve showing a method of an interpretation of dilatograms, basing on which a CHT diagrams were created. It is apparent that during, the first stage of tempering, the investigated steel exhibits shrinkage, which may mainly be attributed to transition iron carbide precipitation. This shrinkage starts at ε_s temperature, and ends at ε_f one. When the ε_f temperature is reached, a positive dilatation effect connected with retained austenite transformation can also be noticed. This effect is noticeable in the temperature range of retained austenite starts (RA_s) ÷ retained austenite finish (RA_f). Both above mentioned effects, especially dilatation effect related to retained austenite transformation, are not intensive. Between $(Cu_\varepsilon)_s$ and $(Cu_\varepsilon)_f$ temperatures the precipitation of copper rich phase (Cu_ε) takes place. **Fig. 4** shows CHT diagram for the investigated steel. The diagram shows the ranges of iron transition carbide and the temperature range for the transformation of the retained austenite as well as the temperature range of precipitation of copper rich phase (Cu_ε). As can be noticed, transformation start and transformation finish temperatures increase with an increasing heating rate from 0.05 to 35 °C/s.

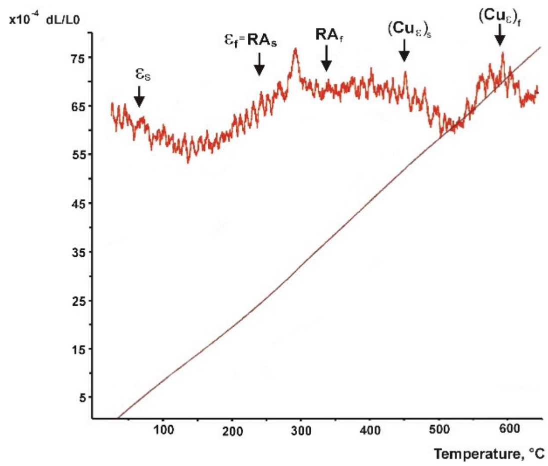

Fig. 3 The dilatometric curve of heating with the rate of 0.5 °C/s as well as the coresponding differential curve with marked interpretation of phase transformation occured in the 17-4PH steel during heating from as-quenched state

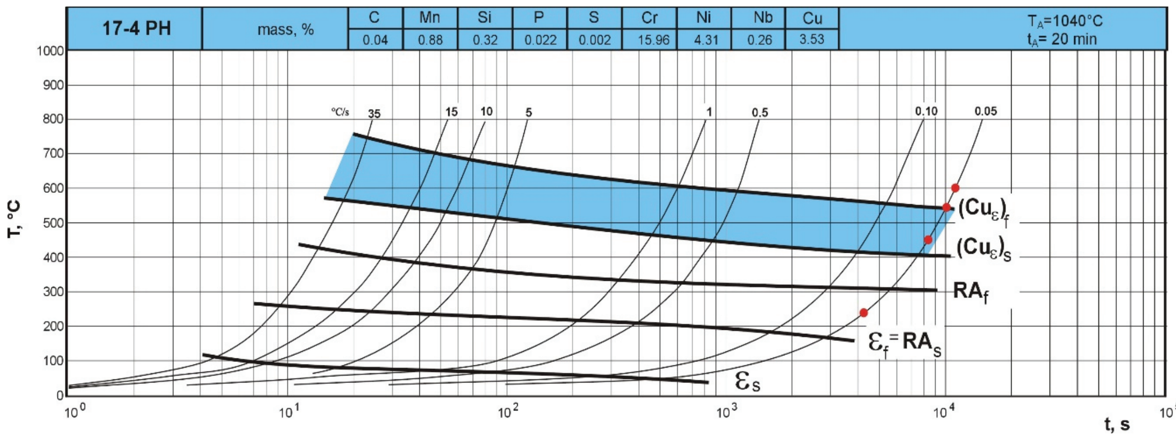

Fig. 4 CHT diagram for investigated steel. The temperatures of the end of heating the samples for metallographic investigation are marked red

Continuous heating from as-quenched state up to 240 °C and 450 °C (**Fig. 5**) does not caused visible changes in the microstructure of investigated steel. But it is worth to note, that heating up to 450 °C increased hardness to 396 HV10. It is results of precipitation starts of copper rich phase (Cu_{ε}) - see **Fig. 4**. Unfortunately we were not able to find it in the microstructure, even by use TEM. Visible changes in the microstructure of the investigated steel were caused by heating it up to 540 °C (**Fig. 6**) and 600 °C (**Fig. 7**). There are clearly visible grain boundaries of a prior austenite and the morphology characteristic for tempered martensite. SEM image presented in **Fig. 6b** and TEM images included in **Figs. 6c and 7c** show precipitates of copper rich phase (Cu_{ε}).

a) b) c)

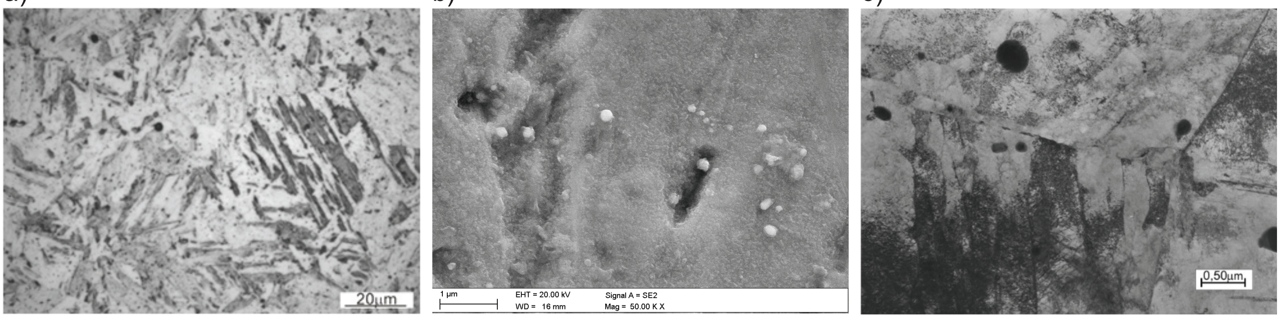

Fig. 5 The microstructure of investigated 17-4PH steel after quenching from 1040 °C and heated up to 450 °C with the rate of 0.5 °C/s: a) light microscope, b) SEM; c) TEM

a) b) c)

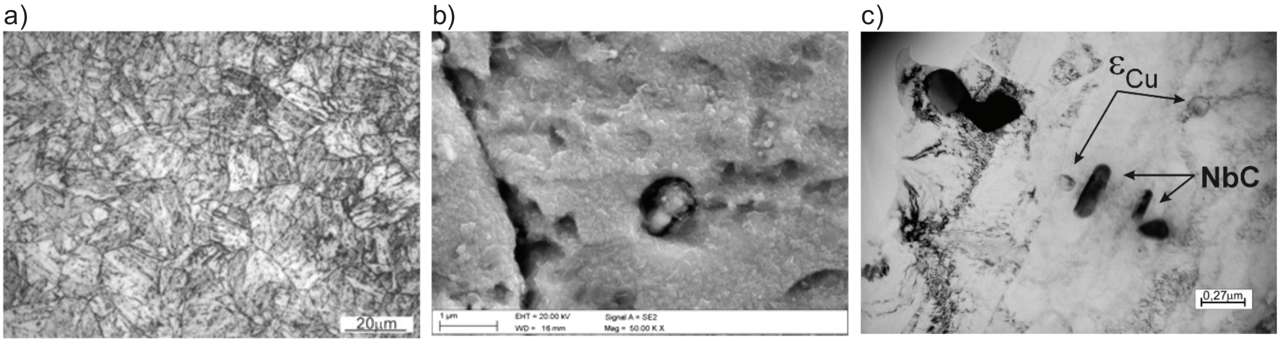

Fig. 6 The microstructure of investigated 17-4PH steel after quenching from 1040 °C and heated up to 540 °C with the rate of 0.5 °C/s: a) light microscope, b) SEM; c) TEM

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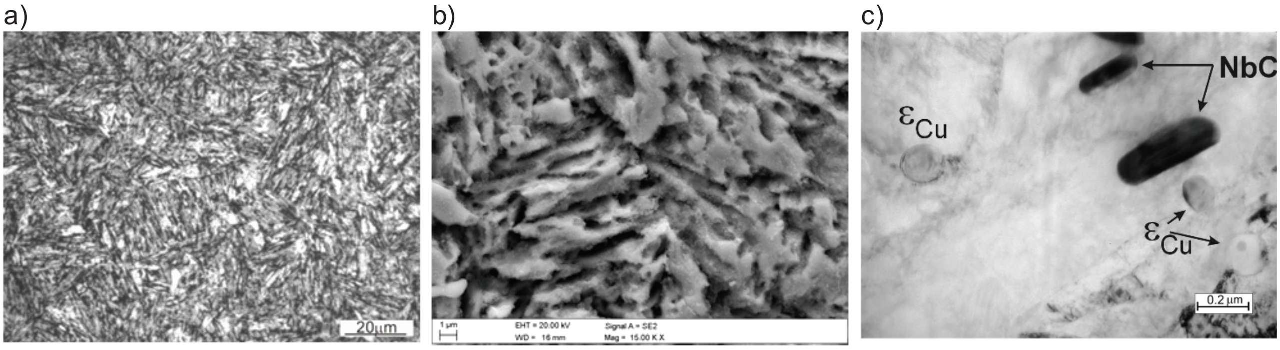

Fig. 7 The microstructure of investigated 17-4PH steel after quenching from 1040 °C and heated up to 600°C with the rate of 0.5 °C/s: a) light microscope, b) SEM; c) TEM

Fig. 8 shows influnce of temperature, up to which sample was heated after quenching (heating rate 0.05 °C/s), on hardness of investigated steel. The highest increase of hardness takes place in the temperature range of

450÷540 °C (in the range of precipitation of copper rich phase). Above 540 °C precipitation of the Cu_{ε} slows down and matrix recovery causes hardness decrease.

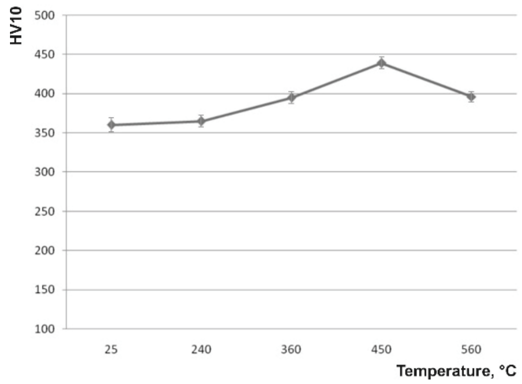

Fig. 8 Dependence of hardness of samples of investigated steel on the heating temperature after quenching (heating rate 0.05 °C/s)

4. CONCLUSIONS

In as-quenched state the microstructure of investigated steel consist of low carbon martensite, ferrite δ banding as well as of not dissolved during austenitizing niobium secondary carbides of MC type. Changes in the microstructure of the investigated steel during heating from the as-quenched state results from precipitation of transition iron carbides, transformation of small amount of retained austenite into martensite and mainly the Cu_{ε} phase precipitation. There were no significant changes in the chemical composition as well as in the morphology of the δ-ferrite.

During continuous heating from as-quenched state the highest hardness increase takes place in the temperature range of 450÷540 °C (in the range of precipitation of copper rich phase). Above 540 °C precipitation of Cu_{ε} slows down and matrix recovery causes hardness decrease.

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