Portevin-Le Chatelier effect in an E911 creep resistant steel with 3%Co additives

A Kipelova¹, R Kaibyshev¹, V Skorobogatykh², I Schenkova²

- ¹ Belgorod State University, Laboratory of Mechanical Properties of Nanoscale Materials and Superalloys, Pobeda 85, 308015 Belgorod, Russia
- ² Central Research Institute for Machine-Building Technology, Sharikopodshipnikovskaya 4, 115088 Moscow, Russia

kipelova@bsu.edu.ru

Abstract. The effect of tempering temperature on mechanical properties of an E911+3%Co creep resistant steel was investigated. The mechanical tensile tests were carried out at temperatures from 298 to 1073 K and at strain rates varying from $2.1 \times 10^{-5} \, \text{s}^{-1}$ to $2.1 \times 10^{-1} \text{s}^{-1}$. The Portevin-Le Chatelier (PLC) effect was found in the temperature range of 473 to 623 K. Various attributes of dynamic strain aging (DSA) like serrated flow with an acoustic emission were observed. With increasing temperature the ultimate tensile strength (UTS) and the yield strength (YS) increased while the ductility decreased. The dependences of the critical plastic strain on strain rate and temperature exhibited "inverse" behavior that was associated with concentrated solid solution in the DSA regime.

1. Introduction

The martensitic steels containing 9-12%Cr are of great interest as components for advanced high efficiency power plants [1, 2]. The beneficial combination of mechanical properties of these steels is achieved by a complex alloying to provide both solution and precipitation strengthening. The latter is resulted from homogeneous distribution of stable second-phase nanoscale dispersoids.

Dynamic strain aging (DSA) was shown in martensitic/ferritic steels due to the interaction of interstitial atoms such as C and N with mobile dislocations during plastic deformation under specific conditions of temperature and stain rate. The DSA results in serrated stress-strain curves, promotes a work hardening, which increases an ultimate tensile strength, and reduces a ductility [3, 4]. Concerning applications, the strengthening effect of DSA can be used to improve mechanical properties such as strength, fatigue and creep resistance [5]. On the other hand, the DSA resulted in several detrimental consequences, i.e. decreased ductility and toughness, and increased the fracture appearance transition temperature. The aim of the present work is to examine the influence of tempering temperature on the temperature range of the PLC effect and its influence on mechanical properties of an E911+3%Co creep resistant steel.

2. Experimental procedure

The chemical composition of investigated E911+3%Co creep resistant steel was (C:0.13, Cr:8.6, Co:3.2, W:1.2, Mo:0.9, V:0.2, Cu:0.1, Nb:0.07, Si:0.06, Ni 0.05, N:0.04, Mn:0.02, B:0.005, all in

mass%, and the balance Fe). The samples were austenitized at 1323 K for 0.5 h followed by air cooling. Then, the specimens were tempered at temperatures ranging from 473 to 1073 K for 3 hours followed by mechanical tests at the same temperatures. The tensile tests were carried out at strain rates of 2.1×10^{-5} s⁻¹ to 2.1×10^{-1} s⁻¹ by using an Instron model 5882 universal testing machine. Tensile specimens of 3×1.5 mm² cross section and 12 mm gauge length were used.

Differential scanning calorimetry (DSC) measurements were conducted on heating and cooling between 298K and 1323 K at the rates of 2 and 10 K/min to analyze a precipitation behavior of the steel. The DSC disk samples with a diameter of 5 mm and a thickness of 0.4 mm were heated and cooled in Al₂O₃ crucibles in an argon atmosphere.

The tempered microstructures was examined by using a JEM-2100 transmission electron microscope (TEM) operating at 200 kV that was equipped with an INCA energy-dispersive X-ray spectrometer (EDS). The TEM foils were prepared by the double jet electro-polishing method using a 10% solution of perchloric acid in glacial acetic acid. Extraction replicas for the microchemical analysis of precipitates were prepared by the evaporation of carbon onto a polished and etched sample surface followed by dissolution of the metallic matrix in a 10%HCl with ethanol at a voltage of 2 V at 293 K. The extraction replicas were caught on small copper grids and cleaned with ethanol.

3. Results and discussion

Serrated stress-strain curves associated with DSA were observed at temperatures of 473 to 623 K and at strain rates varying from 2.1×10^{-5} s⁻¹ to 1×10^{-1} s⁻¹ (figure 1(a), (b)). The type B serrations occurred

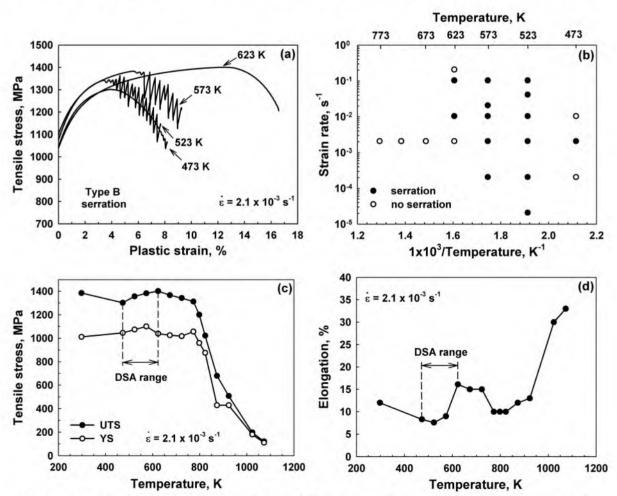


Figure 1. Mechanical properties of an E911+3%Co steel: typical strain-stress curves representing type B serrations (a); temperature-strain rate interval of serrated flow (b); temperature dependence of strength (c) and ductility (d).

under conditions of DSA regime. Type B serrations are oscillations about the general level of the stress-strain curve that occur in quick succession due to discontinuous band propagation arising from the DSA of the moving dislocations within the Luders band [6]. Serrations appear when the carbon or nitrogen concentration on the dislocation exceeds the critical concentration required for the formation of sufficiently large atmosphere. This phenomenon involves the pipe diffusion of carbon or nitrogen from forest dislocations to the waiting mobile dislocations [3].

The temperature dependences of UTS, YS and ductility are shown in figures 1(c), (d). In the DSA range the UTS and the YS gradually increased with increase in the test temperature to 623 K and 573 K, respectively (figure 1(c)). At the same, the ductility exhibited non-monotonous behavior with a minimum at 523 K (figure 1(d)). This suggests that the Luders bands can be potential sites for failure during serrated flow, deteriorating the ductility; and the probability of such failure increases as the material work-hardens. In the DSA range an "inverse" behaviour [7] of the critical plastic strain with strain rate and temperature was found (figure 2(a), (b)). The strain rate and temperature dependencies of critical plastic strain are connected with concentrated solid solution.

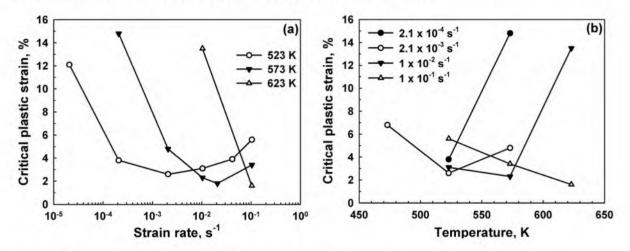


Figure 2. Dependence of the critical plastic strain on strain rate (a) and temperature (b).

The weak exothermic reactions were detected by DSC on heating with the rates of 2 and 10 K/min at 483 and 623 K, respectively. These reactions correspond to the precipitation of second phase particles. The quenched steel samples contained occasional equiaxed particles, approximately 30 nm in diameter; they were identified as NbC by EDS spectra and electron diffraction of the particles extracted from the matrix. Two types of particles, namely, equiaxed NbC particles with an average size of about 18 nm and elongated (Fe, Cr)₃C particles of approximately 80 × 30 nm in size that uniformly distributed within the matrix were observed in the steel tempered at 573 K (figure 3(a)). The ratio of Cr/Fe less than 0.2 was found in the (Fe, Cr)₃C particles. The interaction of these carbides with moving dislocations is clearly seen in the TEM micrograph taken from the deformation substructure after tensile test (figure 3(b)). The volume fraction of homogeneously distributed NbC and (Fe, Cr)₃C carbides was increased with increase in the tempering temperature to 773 K. The amount of Cr in (Fe, Cr)₃C carbides was almost fivefold increased. The increased number of second phase particles led to an increase in the YS and a decrease in the ductility at temperatures of around 773 K (figure 1(c), (d)). Disappearance of DSA at temperatures above 623 K was discussed as a result of the numerous precipitations of fine uniformly distributed carbides acting as extremely effective sinks depleting the interstitial (carbon/nitrogen) atmosphere at dislocations [3]. Further increase in tempering temperatures up to 1073 K led to additional precipitation of Cr₂₃C₆ carbides. The intensive carbides precipitation in the temperature range of 773 to 1073 K reduces concentration of alloying elements in solid solution and terminates the DSA. It was also suggested that at elevated temperatures and low

strain-rates, the blocked dislocations could drag their atmosphere with them and serrated yielding was unlikely to occur [3].

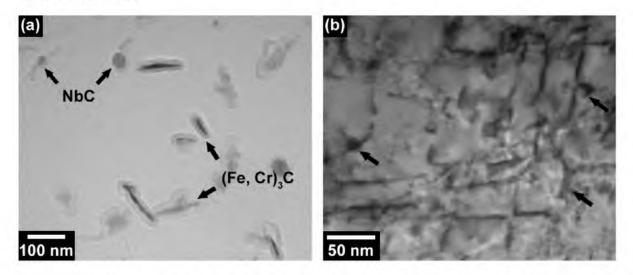


Figure 3. TEM micrographs of the E911+3%Co steel after austenitizing at 1323 K for 0.5 h and tempering for 3 h at 573 K (a) and additionally deformed at 573 K (b).

4. Conclusions

The influence of tempering temperature on the temperature range manifestation of the PLC effect and its effect on the mechanical properties of an E911+3%Co creep resistant steel was examined. The main results are as follows:

- 1. The E911+3%Co steel demonstrates occurrence of PLC effect in the temperature range of 473 to 623 K due to phenomenon of DSA with the following typical attributes: serrated plastic flow with an acoustic emission, increasing the UTS and the YS along with decreasing the ductility as temperature increases.
- 2. Intensive carbide precipitation at temperatures above 623 K is responsible for the disappearance of DSA during the tensile tests at elevated temperatures.

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