Materials Characterization 132 (2017) 437–447

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Recrystallization, precipitation and flow behavior of D3 tool steel under hot working condition

S. Zangeneh Najafi[a], A. Momeni[a,*], H.R. Jafarian[b], S. Ghadar[a]

[a] Materials Science and Engineering Department, Hamedan University of Technology, Hamedan, Iran
[b] School of Metallurgy and Materials Engineering, Iran University of Science and Technology, 16846-13114 Tehran, Iran

ARTICLE INFO

Keywords:
Hot working
Flow curve
Continuous dynamic recrystallization
Particle stimulation of nucleation
Electron back-scattered diffraction

ABSTRACT

Hot compression tests were performed on D3 cold-work tool steel at various temperatures of 800–1200 °C and strain rates of 0.001–1 s^{-1}.The peak stress and strain of flow curves showed uncommon increase within the temperature range of 800–1000 °C and strain rates of 0.001–0.1 s^{-1}. The dynamic precipitation of fine carbides was found responsible for the observed irregularities. Optical microscopy observations showed the highest rate of precipitation at about 1000 °C. At high strain rates (0.1 or 1 s^{-1}), the precipitation temperature shifted to around 1100 °C. Observations by optical and FESEM microscopes and EBSD measurements implied that dynamic precipitation could retard dynamic recrystallization at low temperatures and strain rates. Partial dynamic recrystallization mainly occurs by "continuous dynamic recrystallization" and partly by "discontinuous dynamic recrystallization" and "particle stimulated nucleation" mechanisms. More dissolution of carbides, less dynamic precipitation and considerable continuous dynamic recrystallization were the major microstructural phenomena at high temperatures (1100 °C and 1200 °C). The material constants in the hyperbolic sine constitutive equation were determined for low and high temperature regimes. The apparent activation energy for low and high temperature regimes was determined as 566 kJ/moland 564 kJ/mol, respectively.

1. Introduction

Hot deformation is a practical technique for refining the microstructure of many industrial alloys, including tool steels. The evolution of microstructure during hot working has a crucial effect to the mechanical properties of final products. High-temperature thermomechanical treatment (HTMT) is a common technique for the processing of tool steels [1,2]. Most of plain carbon and low alloy steels are preheated to the austenite phase domain and easily deformed without any concern about transformation or precipitation during hot working. However, the high content of alloying elements and carbon which are added to improve strength, hardness, hardenability, wear resistance and cutting ability in tool steels arise the tendency for precipitation of carbides during solidification and deformation. Dynamic precipitation (DP) during hot working increases flow stress and decreases ductility and deteriorates hot workability [3–5]. Most of the researches devoted to the topic have shown that controlling the microstructural evolutions such as recrystallization, precipitation and grain growth during HTMT is crucial to the required attributes in toll steels [6–10].

Dynamic recovery (DRV) and recrystallization (DRX) are the major softening mechanisms of a material which is subjected to hot working [6,11]. Materials with high stacking fault energy (SFE), such as ferritic steels, are recovered easily, giving rise to equilibrium between work hardening and DRV. The stress–strain curves of these metals, in hot working, show a progressive increase in flow stress to reach a steady state plateau. In contrast, in austenitic steels, intrinsic low SFE restricts dislocations climb and cross slip, thereby diminishing the amount of recovery [6,11–13]. In such materials, the dislocation substructure becomes denser and more inhomogeneous through straining and the accumulative stored energy reaches a critical value for the nucleation of new grains by DRX [6,13,14]. The softening by DRX results in an abrupt decline in flow stress after the work-hardening, leaving a peak on the flow curve [1,15].

Depending on the materials nature and deformation variables, DRX nucleation occurs by either a continuous or discontinuous mechanism (CDRX and DDRX). In DDRX which is commonly observed in low SFE metals, strain-induced boundary migration (SIBM) leads to the formation of serrations and bulging on the initial grain boundaries. The well-developed bulges eventually form the DRX nuclei [16–19]. In contrast, CDRX happens when pretty mature subgrains, formed during extended DRV, turn into the new DRX grains [2,18]. Actually, CDRX occurs in materials having high SFE values or when the occurrence of DDRX is

* Corresponding author.
E-mail address: ammomeni@aut.ac.ir (A. Momeni).

http://dx.doi.org/10.1016/j.matchar.2017.09.009
Received 18 June 2017; Received in revised form 9 September 2017; Accepted 11 September 2017
Available online 14 September 2017

hindered by vast DP [20]. The predominant mechanism of DRX not only depends on the intrinsic material properties, such as SFE, but also varies with the deformation variables of strain rate and temperature [2,21].

The role of DP in hot working is not limited to the restriction of DRX [11,18,22]. It has been approved that large second-phase particles may even help DRX by the mechanism so-called "particle stimulation of nucleation" (PSN) [6,12,18,23]. The contribution of PSN to DRX in tool steels is expected when large chromium carbide particles are present in hot working temperatures.

Despite the extensive researches devoted to the hot deformation behavior of different kinds of steels, less attention has been paid to the cold work tool steels. This can be attributed to the sophisticated hot working behavior due to the high alloying element contents. It is thought that all the mentioned phenomena such as DRV, CDRX, DDRX, DP and PSN possibly happen during hot working of tool steels. By the way, the previous publications have shown that we should take into account the effect of pre-existing carbide particles which tend to spheroidize or break down during hot working [24,25]. On this basis, current investigation has aimed at examining the complexities regarding numerous underlying mechanisms governing the hot deformation behavior of D3 cold-work tool steel which is one of the most familiar grades of tool steels in different industries.

2. Experimental Procedures

The material used in this investigation is D3 (= X210Cr12, comparatively AISI D3) high Cr- high C cold work tool steel, with chemical composition, in weight percent, according to Table 1.

The cylindrical compression samples of 15 mm height and 10 mm diameter were prepared from the as-received hot forged bar according to ASTM E209 standard. Before testing, all the specimens were preheated to 1200 °C, held for 15 min, followed by cooling down to testing temperatures. After soaking for 3 min, continuous hot compression tests were carried out at the temperatures of 800 °C, 900 °C, 1000 °C, 1100 °C and 1200 °C with strain rates of 0.001, 0.01, 0.1 and 1 s^{-1}. In order to preserve the as-hot worked microstructure, the hot deformed samples were quenched within 3 s after unloading. Before using the experimental flow curves they were corrected for the effects of adiabatic heating and friction using the methods proposed in the literature [26,27].

In order to characterize the microstructure of samples before starting the hot compression tests a witness sample was reheated to 1200 °C, held for 15 min and then quenched to the ambient temperature. Fig. 1 schematically demonstrates the thermomechanical cycle that the specimens were subjected to in this research and the microstructure of the witness sample before starting the hot compression tests. The volume fraction of initial carbides observed in Fig. 1 (b) was measured by the image analyzer software as about 8.6%.

The hot deformed samples were cut along the longitudinal axes and prepared by the standard metallographic techniques for microstructural observations. A reagent composed of 1 g picric acid, 1 ml HCl and 78 ml distilled water was used to reveal the prior austenite grain boundaries in the deformed samples. Besides, in order to outline carbide particles a two-stage etching procedure was adopted as: a) primary etching in a solution composed of 9 ml glycerin, 6 ml HCl and 3 ml HNO_3 for 4 s, and b) final etching in the Marble solution composed of 4 g $CuSO_4$, 16 ml HCl and 30 ml distilled water for 25 s. This procedure caused a binary microstructure of bright carbide particles within dark matrix appropriate for quantification by MIP image analyzer program.

Table 1
Chemical composition of D3 cold work tool steel used in the present investigation.

C	Si	Mn	Cr	Mo	Ni	Co	Cu	P	Fe
1.79	0.09	0.43	11.50	0.05	0.24	0.02	0.18	0.03	Balance

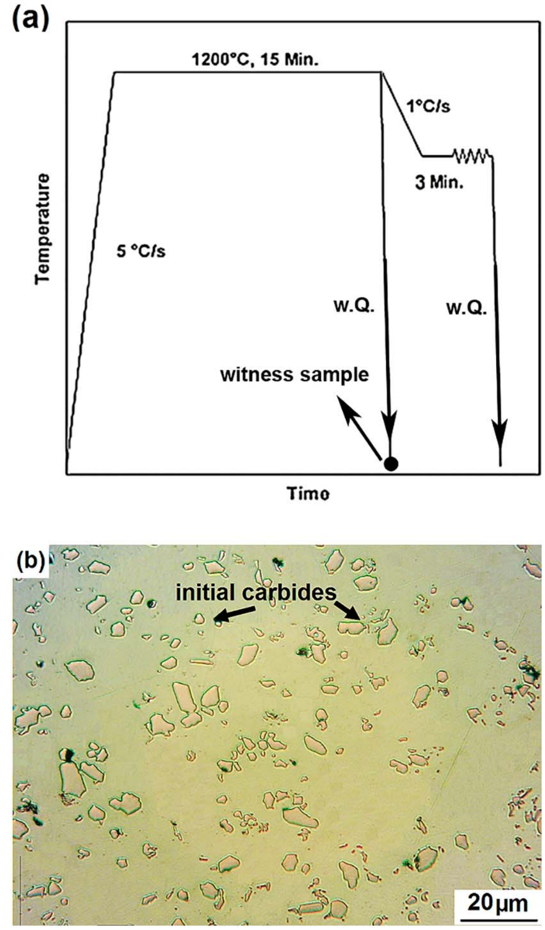

Fig. 1. (a) Thermomechanical treatment carried out on the samples in this research and (b) microstructure of the witness sample after reheating to 1200 °C for 15 min and quenching in water.

The microstructures of hot deformed samples were primarily characterized by optical microscope and carbides volume fraction was determined by MIP image analysis software. For each deformation condition the volume fraction of carbides was determined from four measurements. Field emission scanning electron microscope (FE-SEM Philips XL30S-FEG) equipped with the electron back-scattering diffraction (EBSD) detector was utilized to study the microstructural evolutions with more details. The sections of the samples for EBSD observation were mechanically polished followed by electropolishing in a 90% CH_3COOH + 10% $HClO_4$ at voltage of 20 V for 20 s at room temperature. TSL OIM software was used to analyze the EBSD data.

3. Results and Discussion

3.1. Flow Stress Analysis

The stress–strain curves for the studied material are shown in Fig. 2. As expected, the flow stress level increases as the strain rate rises or temperature declines. In the whole range of temperature and strain rate, the flow curves exhibited the typical behavior of DRX, i.e. strain hardening to a peak stress, σ_p, and then work softening towards a steady state stress, σ_{ss}. In the steady-state region, there is a dynamic balance between work hardening and flow softening. The peak point of a typical DRX flow curve is generally shifted to higher strains and stresses as temperature declines or strain rate rises [1,5,6]. However, the peak points, arrowed in Fig. 2, do not show a clear variation with temperature or strain rate. The irregularities in the position of the peak is more pronounced at 900–1100 °C, so that the peak strain, ε_p,