Materials Science & Engineering A 596 (2014) 244–249

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

Strengthening mechanism and thermal stability of severely deformed ferritic/martensitic steel

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ARTICLE INFO

Article history:
Received 23 May 2013
Received in revised form
30 November 2013
Accepted 16 December 2013
Available online 24 December 2013

Keywords:
Ferritic/martensitic steel
Severe plastic deformation
Equal-channel angular pressing
Internal friction

ABSTRACT

The strengthening mechanism of the ultrafine-grained ferritic/martensitic steel (T91) prepared by equal-channel angular pressing (ECAP) is studied by microhardness measurement, transmission electron microscopy and internal friction. The results show that dislocation strengthening rather than grain boundary strengthening plays a dominant role in the increase in microhardness. Upon annealing at different temperatures, it is found that there is an evident decrease in microhardness between 650 and 700 °C for the ECAPed specimens processed by 1 pass and 4 passes. An internal friction peak is also observed in the vicinity of 670 °C for the ECAPed specimens during the first heating but disappears during the second heating. The TEM observations indicate that there is no evident change in the grain size for the ECAPed specimens before and after the annealing at 700 °C for 1 h. It is concluded that the softening around 670 °C stems mainly from the dislocation annihilation.

1. Introduction

Refining grain size of materials into ultrafine-grain (UFG) or nanocrystalline (NC) scale can generally improve the mechanical properties (e.g., strength or hardness) of materials. Severe plastic deformation (SPD) methods have been proved to possess remarkable ability to produce UFG/NC metals and alloys [1]. In general, the improvement in strength originating from the grain refinement is a trade-off with ductility (for example, UFG ferritic/martensitic steel [2]), which will restrict practical application of the UFG/NC materials as engineering materials. However, some recent experimental results have demonstrated that both solely SPD processing and combining SPD processing with alloying can simultaneously enhance the strength and ductility of tungsten, bronze and Cu–Al alloys [3–5].

Under SPD processing, various defects such as point defects and dislocations will be introduced into metals. Since the point defects are readily annealed out at lower temperatures and have little effect on the mechanical properties of the materials [6], the dislocation behaviors determine the microstructure evolution and the changes in mechanical properties of the metals subjected to the SPD processing. To minimize strain energy, the dislocations introduced into the grain interiors during SPD have propensity to arrange to the cell boundaries (or incidental dislocation boundaries, IDBs) with small misorientations [7,8]. As the deformation proceeds, more and more dislocations accumulate in IDBs to bring about an increase in dislocation density so that the misorientations separating the neighboring cells increase with the thickness of the IDBs, and finally the IDBs transform into the high-angle grain boundaries (geometrically necessary boundaries, GNBs) [7]. Thus, for heavily deformed metals, the main strengthening contributions can be considered from two aspects: (i) dislocation strengthening due to the dislocations in the grain interiors and IDBs and (ii) grain boundary strengthening due to the presence of GNBs [8,9]. Theoretical calculations have predicted that the individual strengthening contribution of IDBs is 50% higher than that of GNBs in Ni when the sample was deformed to a strain value of 4.5 [8]. However, the relative fractions of IDBs and GNBs are very difficult to be estimated experimentally, for example, from X-ray line profile analysis because both boundaries can break down the coherency of X-rays similarly [7]. On another side, there is large uncertainty in achieving the information on grain boundaries and dislocation density by using electron microscopy because their status and quantity will alter during the thinning of specimens. In contrast to the strengthening due to the SPD processing, the softening of SPDed metals may be considered to be a reverse procedure of the strengthening processes, which in principle stems from the annihilation of dislocations and/or coarsening of grain size. Actually, many studies had reported that annealing can soften distinctly the SPDed materials such as T91 steel, pure Cu and Ag [2,9,10]. Nevertheless, the detailed strengthening mechanism of severely deformed metals, namely, which contribution plays a critical role in strengthening, was not clearly understood. The purpose of this study is to investigate the strengthening

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http://dx.doi.org/10.1016/j.msea.2013.12.062

Table 1
The chemical composition (wt%) of the initial T91 steel (Fe in balance).

Cr	Ni	Mo	Mn	Ti	V	Nb	Cu	C	Si	P	S	N
8.63	0.23	0.95	0.43	0.003	0.21	0.09	0.046	0.1	0.31	0.02	0.006	0.03

mechanism and the thermal stability of ferritic/martensitic steel processed by equal channel angular pressing (ECAP, 1–4 passes).

2. Experimental

The ferritic/martensitic steel (T91) was provided by China Institute of Atomic Energy. The final heat treatment was normalized at 1040 °C for 1 h followed by air cooling, and then tempered at 760 °C for 1 h followed by air cooling. The chemical composition is given in Table 1. For this study, the as-received T91 steel was machined to cylindrical rods of ∅10 mm × 60 mm in dimension (see Ref. [2]). ECAP processing was carried out in a die with the intersecting angle of 90°. The rods were extruded in a self-designed ECAP die using 63 t hydraulic press at room temperature. All of the ECAP extrusions were conducted in route C, that is to say, after each extrusion the sample was rotated by 180° around the extrusion direction before the next extrusion. The hardness testing was performed by MH-3 Vickers microhardness tester. Each sample was indented 10 times under a load of 100 g keeping for 10 s. The samples for hardness measurements were heated up to the corresponding annealing temperature with a heating rate of 5 °C/min, and held at these temperatures for 1 h. Then the samples were cooled down to room temperature with a rate of 5 °C/min. Internal friction (IF) was measured by an inverted torsion pendulum with the forced vibration mode in the process of ascending temperature with a heating rate of 5 °C/min. The measurement frequency is in the range of 0.5–11 Hz and the strain amplitude was controlled as 2×10^{-5}. In order to investigate the stability of the IF peak, the IF measurement was repeated in the second run. The microstructure was characterized by transmission electron microscopy (TEM, Jeol) operating at 200 kV. X-ray diffraction (XRD, X′Pert Pro MPD) measurement at room temperature was performed at θ–2θ scan mode with Cu-Kα radiation. Differential scanning calorimetry (DSC) was performed with a thermal analyzer (Perkin-Elmer Pyris Diamond) for a specimen of about 30 mg heated at a rate of 10 K/min.

3. Results and discussion

Fig. 1(a) displays a bright-field TEM micrograph for the as-received T91 steel. The typical grain size of the as-received T91 steel is about 10 μm. The grains consist of tempered martensitic laths with a width of about 0.4 μm. A number of carbide $M_{23}C_6$ (where M is mainly Cr including a little amount of Mo, as confirmed by the EDS measurement equipped in TEM) are distributed on the prior austenite grain boundaries and on the martensitic lath boundaries, which are also reported in reference [11]. After the ECAP extrusion with 1 pass (Fig. 1(b)) and 4 passes (Fig. 1(c)), the carbides $M_{23}C_6$ at boundaries disappeared and the grain sizes were apparently refined to about 400 nm and 300 nm with the equiaxed shape. Also, it can be observed that the dislocation density in the grain interiors in Fig. 1(c) is higher than that in (b). The discontinuous circles in selected area electron diffraction patterns of the ECAP extruded samples shown in the inset of Fig. 1(b) and (c) illustrate as well that the large grains in as-received specimen were refined into a number of small polycrystals with different lattice orientations due to the ECAP extrusion.

The microstructural evolution is usually related to the mechanical properties of materials. To illustrate this relationship the Vickers microhardness was tested and is shown in Fig. 2. The microhardness of the as-received specimen is 240 Hv, which is roughly consistent with the reported data [12], while the microhardness increases to 310 Hv and 320 Hv for the specimens extruded with 1 pass and 4 passes, respectively. This illustrates that ECAP processing can significantly enhance the strength of T91 steel. To investigate the thermal stability, the change of microhardness as a function of annealing temperature within the range of 500 and 850 °C for 1 h is also presented in Fig. 2. For the as-received specimen, the microhardness almost remains unchanged up to 800 °C and thereafter slightly decreases owing to the possible dissolution of carbides after the annealing. For the extruded specimens, the microhardness decreases slightly from 320 Hv to 270 Hv for 500–650 °C annealing and then dramatically decreases for 650–700 °C annealing for both specimens extruded with 1 pass and 4 passes. However, it is worth noting that when the annealing temperature exceeds 700 °C (< 850 °C), the microhardness for the 4 passes extruded samples tend to be a constant as 200 Hv while it continues to decrease slightly to 170 Hv for the 1 pass extruded samples. In this study, the as-received T91 steel was subjected to the standard heat treatment consisting of an austenisation at 1040 °C for 1 h followed by air cooling and subsequent tempering at 760 °C for 1 h. In general, accumulated plastic strain increases with increasing the number of extrusion and high strain will introduce large misorientations within subdivided grains. Therefore, different passes of ECAP will lead to different dislocation structures and subgrain boundaries (i.e., IDBs). It is expected that the dislocations within the grains play a dominant role in microhardness up to 750 °C while the dislocations in IDBs affect the microhardness after 750 °C. The misorientation level resulted in more difficult rearrangement of dislocations in IDBs in the case of the 4 passes extruded samples than in the 1 pass after 750 °C annealing. This may be the reason why the microhardness values in the 1 pass extruded samples is lower than in the 4 passes.

Based on the TEM observations in Fig. 1, since both the increase of dislocation density in the grain interiors and the refinement of grain sizes are simultaneously observed in the ECAPed specimens, it is difficult to distinguish which contribution plays more important role in the change of the hardness. To clarify the strengthening mechanism of severely plastic deformed specimen, the microstructures of the ECAPed specimens subjected to the annealing at 700 °C for 1 h were investigated. Interestingly, as the typical bright-field TEM images shown in Fig. 3(a)–(c), the annealing at 700 °C does not result in the substantial grain growth and the size of most grains remains in the range of 300–400 nm, which is same as that before the annealing (see Fig. 1(b) and (c)). This implies that the GNBs may not dominantly contribute to the strength improvement.

It is well known that grain growth of the nanostructured materials produced by SPD starts at 0.4 T_m and even lower (T_m is the absolute melting temperature) [13]. For the present T91 steel 0.4 T_m is 448 °C (T_m=1803 K [14]) which is much lower than the annealing temperature of 700 °C, suggesting that the UFG T91 steel produced by the ECAP possesses good thermal stability to some extent. In addition, Fig. 3(a) and (b) also shows that the IDBs become more distinct and the width of the grain boundaries