Article

Effect of κ Carbides on Deformation Behavior of Fe-27Mn-10Al-1C Low Density Steel

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Abstract: Fe-Mn-Al-C steel, which is a potential lightweight material for automobiles, has a variety of microstructures and good mechanical properties. The effect of κ carbides on the mechanical properties and strain hardening rate of Fe-27Mn-10Al-1C (wt.%) low density steel was studied by short-time heat treatment to control the precipitation behavior of κ carbides. Quenched specimens have an excellent combination of strength and plasticity and continuous high strain hardening rate, which is due to the uniform distribution of κ carbides with an average size of 1.6 nm in an austenite matrix. The fracture mode of the sample changed from ductile fracture to cleavage fracture, which was because the aging treatment promoted the precipitation of B2 phases and κ carbides at grain boundaries. The size and volume fraction of nanoscale κ carbides in austenite grains increase with the increase of aging temperature, and the yield strength increases but the density of slip bands decreases, resulting in the gradual decrease of strain hardening rate.

Keywords: Fe-Mn-Al-C steel; short-time heat treatment; κ carbides; strain hardening; plane slip

1. Introduction

At present, the automobile industry is seeking to reduce greenhouse gas emissions and improve energy efficiency through lightweight design [1]. Especially for electric vehicles, in addition to the development of high energy density batteries, lightweight design is one of the effective ways to improve the endurance mileage. The density of Fe-Mn-Al-C low density steel is effectively reduced by adding Al into the steel [2–4], which can be used as one of the lightweight structural materials for automobile body. Compared with other lightweight materials (such as magnesium alloy, aluminum alloy, carbon fiber composites, etc.), Fe-Mn-Al-C low density steel has been widely studied because of its high strength, high plasticity, high specific strength, serviceability in corrosive environments and excellent performance combination at low cost [5–16].

It has been reported that the formation of L’12 type ’Fe(Mn)3AlC phase (κ) can be promoted by holding Fe-Mn-Al-C steel with high Mn and Al content at 450–550 °C for several hours. [17–23] and the tensile strength can be improved by a precipitation hardening mechanism. [17,18,24,25]. Therefore, scholars have carried out extensive studies on the effect of aging treatments on microstructure and properties [8–10,16]. However, in the heat treatment process used in previous studies, the solution treatment (ST) time is mostly several hours, and the subsequent aging time is as long as tens of hours [8–10,16,26]. That is to say, a heat treatment time that is too long has limited performance improvement but increased energy consumption. The precipitation behavior of κ carbides not only enhances the strength but also plays an important role in the plastic deformation process. Scholars have studied the effects of different Al content [16] and different aging time [27] on the strain hardening rate, but there are few studies on the deformation behavior at different aging temperatures under the conditions of short-term heat treatment.
Therefore, in this work, the time of heat treatment was significantly shortened on the basis of previous studies for industrial applications. The effect of precipitation behavior of the κ carbide on deformation behavior of Fe-27Mn-10Al-1C (wt.%) steel under short-term heat treatment was studied.

2. Materials and Methods

A 50 kg ingot of a Fe–27Mn–10Al–1C steel was prepared by induction melting in an argon atmosphere. The chemical composition of the steel was measured as 0.98C, 26.5Mn, 10.1Al, 0.45Si, 0.17V and the remainder as Fe (wt.%). According to the empirical formula [2,5], the density of the experimental steel is about 6.5 g/cm³, which is about 16% lower than that of the ordinary steel. After homogenization at 1200 °C for 2 h, the ingot was hot rolled to 4.0 mm thick steel plate at 1050 °C. The hot rolled steel plate was annealed at 1000 °C for 1 h to eliminate the deformation resistance and then cold rolled to the final thickness of about 1.6 mm. The cold rolled steel plate was solution treated at 1000 °C for 5 min and then quenched in water. The sample was named S1000. The S1000 samples were aged at 450, 500 and 550 °C for 90 min, respectively, and then quenched in water. The samples after three aging treatments were named as A450, A500 and A550, respectively. The dog-bone specimens with a standard distance of 25 mm and a width of 6 mm were used for tensile tests at room temperature at a deformation rate of 2 mm/min. The sample surface was polished, and the tensile axis was parallel to the rolling direction. The microstructure and fracture morphology after heat treatment were observed by Hitachi SU2500 field emission scanning electron microscope (SEM). The phase identification of heat-treated samples was performed using a Panalysis X-ray diffractometer (XRD). The microstructure after tensile deformation was observed by a Tecnai G2F30 field emission transmission electron microscope (TEM).

3. Results

3.1. Tensile Properties

Figure 1a shows the stress–strain curves of S1000, A450, A500 and A550 specimens. The values of 0.2% yield strength (YS), ultimate tensile strength (UTS) and elongation (El) after fracture are plotted in Figure 1b. The steel after solid solution treatment showed extraordinary mechanical properties, in addition to the high ultimate tensile strength of 916 MPa and the yield strength of 710 MPa, it also had 56% ultra-high plasticity. The yield strength of the samples aged at different temperatures was significantly increased (about 80 MPa, 240 MPa and 330 MPa, respectively), while the ultimate tensile strength was slightly increased (about −40 MPa, 40 MPa and 130 MPa, respectively). The difference between ultimate tensile strength and yield strength of the specimen gradually decreased (about 80 MPa, 5 MPa, 6 MPa, respectively) and the values became closer and closer.

The true stress–strain curves of specimens S1000, A450, A500 and A550 are shown in Figure 1c. The plastic deformation processes of the four specimens are all continuous yielding. When the stress reaches the critical yield stress value, the true stress of the specimen increases with the increase of true strain until fracture failure, showing a linear relationship and continuous work hardening behavior.

The strain hardening rate–true strain curves of S1000, A450, A500 and A550 samples are plotted in Figure 1d. It can be easily seen that in the whole plastic deformation zone, with the increase of real strain, the specimen exhibits three stages of strain hardening behavior. That is to say, the strain hardening rate decreases rapidly in the first stage, remains unchanged in the second stage, and then decreases again in the third stage with the increase of real strain. It is worth noting that the value of strain hardening rate which remains unchanged in the second stage decreases with the increase of aging temperature.

From the current results, increasing aging temperature significantly improves the yield strength of steel but reduces the strain hardening ability.
Figure 1. The tensile properties of samples S1000, A450, A500 and A550 plotted: (a) room temperature tensile stress–strain, (b) values of the YS, UTS and EI, (c) true stress–strain and (d) strain hardening rate curve.

3.2. Fractography

The fracture morphology of specimens S1000, A450, A500 and A550 after the room temperature tensile test was observed to characterize their fracture behavior. Figure 2a shows the SEM images of specimen S1000 after tensile deformation and fracture. It is observed that the fracture mainly presents the dense distribution of small dimples. Figure 2b–d are SEM photographs of A450, A500 and A550 specimens after tensile deformation and fracture. Large dimples were observed in A450 sample, and slip bands were also observed in A500 and A550 samples in addition to dimples.

3.3. Microstructure of Solid Solution Treated and Aged Specimens

Figure 3a is the SEM photos of the sample S1000, and the microstructure of the sample S1000 is mainly austenite and banded ferrite. Figure 3b–d showed the SEM images of A450, A500 and A550, respectively. The microstructure of the samples was observed to be composed of austenite, ferrite and inter-grain precipitation. It was also observed that precipitates were easier to accumulate at ferrite and austenite grain boundaries. The number of precipitates increases with aging temperature. According to the XRD results shown in Figure 3e, these precipitates were identified as κ carbides with FCC structure and B2 phases with BCC structure.
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3.4. Ordered Phase

Figure 4a is the TEM image of S1000 specimen after tensile deformation. It can be observed that the dislocation slip movement in place bypasses the hard particles. Figure 4b,c shows the selected-area diffraction (SAD) patterns in the selected area of the matrix and the hard particles, respectively. The matrix and the hard particles are determined as austenite and B2 phases. Figure 4d,f shows that B2 phases is rich in aluminum but poor in iron and manganese. In addition, Figure 4c shows that DO3 phases exists in B2 phases.
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Figure 4. TEM analysis of the γ and B2+DO3 phases in S1000 samples: (a) TEM image, (b,c) SAD pattern analysis and (d-f) EDS mapping for Fe, Mn and Al.

Figure 5 shows the TEM weak beam dark field (WBDF) images of samples S1000, A450, A500 and A550. Figure 5a shows that κ carbides distribute uniformly in austenite matrix after solution treatment. The average size and volume fraction of κ carbides in sample S1000 were 1.6 nm and 4.7%, respectively. The average size and volume fraction of sample A450 were 2.8 nm and 9.6%. The average size and volume fraction of sample A500 are 3.7 nm and 13.3%. Sample A550 was further increased to 4.5 nm and 16.1%. Increasing aging temperature promotes the increase of size and volume fraction of κ carbides.

3.5. Microstructure after Tensile Deformation

Figure 6 shows the TEM images of specimens S1000, A450, A500 and A550 after tensile deformation. S1000 sample shows fine slip bands at the end of deformation (Figure 6a). In A450 sample, slip bands intersect to form Taylor lattice structure (Figure 6b). In the A500 sample, the spacing of slip bands increases further (Figure 6c). The A550 sample is a typical microstrip structure with high density dislocation wall structure (Figure 6d). It is not difficult to see that the spacing of slip bands increases with the increase of aging temperature. In other words, the density of slip bands decreases.
Figure 5. TEM weak beam dark field (WBDF) images of (a) S1000, (b) A450, (c) A500 and (d) A550 specimens.

Figure 6. TEM images of (a) S1000, (b) A450, (c) A500 and (d) A550 specimens after stretching and deformation.

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### 4. Discussion

#### 4.1. Precipitate Strengthening of κ Carbides and B2 Phases

As shown in Figure 5a even in an as-quenched state, a high density of nanosized κ′-carbides are formed and uniformly distributed within the austenitic matrix by spinodal decomposition during quenching [28,29], which is attributed to the high additions of Al and C [30,31]. With the increase of aging temperature, the average size and volume fraction of κ carbides in austenite grains gradually increase (Figure 5b–d) and the precipitation of κ carbides and B2 phases at grain boundary increase (Figure 3b–d), which is due to the transformation of solute-rich solid solution into solute-poor solid solution, κ carbides and B2 phases after aging treatment. We also note that even in solid solution-treated samples, sub-micron B2 phases are found in the austenite matrix and hinder the dislocation slip movement (Figure 4). Wang et al. prepared Fe-Mn-Al-C steel with double nano-precipitation of hard B2 phase and κ carbide by short-time annealing at 800–900 °C. The ultimate tensile strength of the steel was measured to be more than 1.7 GPa, and the elongation was more than 10% [32]. It can be seen from Figure 5a that the uniform distribution of nano-sized κ carbide and sub-micron hard B2 particles in the austenite matrix is that the S1000 sample maintains the ultimate tensile strength of 916 MPa and the elongation reaches 56%. This short-time heat treatment is very easy to obtain tens to hundreds of nanometers of hard B2 phase in austenite matrix. Table 1 lists the yield strength, tensile strength and elongation of Fe-Mn-Al-C steel with similar composition under long-time heat treatment. It can be seen from the comparison that the strength and elongation of the test steel reached the average or even superior level under the short-term heat treatment process. There are two main reasons for the significant increase in the yield strength of the aged specimens with the increase of aging temperature. One is the increase in the average size and volume fraction of the ordered phases κ carbides in the austenite grain. The other is the formation of a large number of fine κ carbides and B2 precipitates at the grain boundary. In other words, the synergistic effect of intracrystalline precipitation and grain...
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Table 1. Mechanical properties of several Fe-Mn-Al-C steels with similar composition after long-time heat treatment.

| Materials          | ST Aging | YS/MPa | UTS/MPa | El/% | References |
|--------------------|----------|--------|---------|------|------------|
| Fe-30Mn-9Al-1.5Si-0.5Mo | 1050 °C + 2 h, 530 °C + 10 h | 891 | 940 | 18 | [8] |
| Fe-30Mn-9Al-1.4Si-0.9C-0.5Mo | 1050 °C + 2 h, 530 °C + 10 h | 922 | 995 | 14 | |
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| Fe-30Mn-9Al-1.4Si-0.9C-0.5Mo | 1050 °C + 2 h, 530 °C + 10 h | 922 | 995 | 14 | |
| Fe-26Mn-8Al-11Si-0.90C-0.53Mo | 1050 °C + 2 h, 530 °C + 10 h | 1016 | 1085 | 5 | [9] |
| Fe-26Mn-8Al-11Si-0.90C-0.53Mo | 1050 °C + 2 h, 530 °C + 10 h | 1016 | 1085 | 5 | [9] |
| Fe-26Mn-10Al-1C | 1000 °C + 15 min, 550 °C + 40 h | 873 | 953 | 20.1 | |
| Fe-26Mn-10Al-1C | 1000 °C + 15 min, 550 °C + 40 h | 873 | 953 | 20.1 | [16] |
| Fe-29.8Mn-7.6Al-1.11C | 1150 °C + 5 h, 550 °C + 16 h | 880 | 995 | 26 | [10] |

4.2. Fracture Behavior

O. Acselrad et al. analyzed the dynamic impact fracture behavior of Fe-Mn-Al-C steel and proposed that the slip zone fracture was caused by a plane slip and the main fracture mechanism was slippage along the slip surface [12]. It can be seen from Figure 2a that fine and dense dimples can be observed in the fracture of specimen S1000, showing the ductile fracture characteristics with dimples. With the increase of aging temperature, the fracture morphology of different aging samples is also different. The dimples of sample A450 become larger, and A500 and A550 have slip bands besides dimples. This change is due to the gradual increase of $\kappa$ carbides and B2 phases at grain boundary with the increase of aging temperature. They precipitate at the grain boundary, causing local stress concentrations and then generate microcracks. These microcracks propagate along the
grain boundary during plastic deformation and eventually lead to the fracture of specimens A500 and A550 along the grain boundary [33]. The plasticity was seriously deteriorated by precipitation of κ carbides and B2 phases at grain boundary, and the elongation decreased from 56% of the S1000 sample to 24% of the A550 sample. Therefore, although this short-time aging treatment significantly improves the yield strength, the effect on reducing plasticity is also very obvious.

4.3. Reduction of Strain Hardening Rate

In fact, it is reported that the plane slip of austenitic Fe-Mn-Al-C steel is the dispersion of nanoscale shearable κ carbides in the austenitic matrix, leading to the softening effect of slip surface. [27,34,35]. In the early stage of plastic deformation of low density steel, the initial dislocation moves on the slip surface to shear the κ carbides, which destroys the short range order (SRO) structure, and then the dislocation movement on the same slip surface encounters less resistance, which leads to the softening effect of the slip surface. We also observed that sub-micron κ-carbide was sheared along the slip band in TEM images of A500 samples, as shown in Figure 7. One of the strengthening mechanisms of Fe-Mn-Al-C steel is the refinement effect of dynamic slip bands, that is, during plastic deformation the spacing of slip bands decreases with the increase of strain and eventually high-density slip bands are formed. Ref. [36] (Figure 6a), shows continuous high strain-hardening ability. The size and volume fraction of κ carbides increase with the increase of aging temperature (Figure 5) leading from weak barriers to dislocation motion and reducing the boundaries of the slip bands for accumulation dislocations during plasticity deformation, which results in the lowering of the strain hardening rate [37]. Therefore, it is not difficult to conclude that, in addition to the softening effect caused by the shear of κ carbide in austenite grain, with the increase of aging temperature, even if the increase of κ carbide size and volume fraction is small, it still leads to the decrease of slip band density, which leads to the decrease of strain hardening rate.

Figure 7. TEM images of (a,b): the κ carbide of A500 sample was sheared by the slip band.

5. Conclusions

In this work, the effect of κ carbides on the mechanical properties and strain hardening rate of Fe-27Mn-10Al-1C low-density steel was studied by changing the aging temperature to control the precipitation behavior of κ carbides. The conclusions obtained from the experiments are as follows:

(1) Submicron B2 phase was uniformly distributed in the austenite matrix of Fe-27Mn-10Al-1C steel prepared by a short-time solid solution treatment and the matrix was strengthened by the Orovan mechanism.

(2) After short-time aging, the size and volume fraction of κ carbide in austenite grain, the precipitation amount of B2 phase and the κ carbide at grain boundary increased with the increase of aging temperature. The yield strength was increased by 80 MPa, 240 MPa and 330 MPa, respectively. The yield strength of sample A550 is more than 1.0 GPa, and the
good elongation is maintained at 24%. The performance is comparable to that of long-time aging steel.

(3) Short-time aging failed to avoid the precipitation of κ carbide and B2 phase at grain boundary of Fe-27Mn-10Al-1C steel, and the fracture mode of the sample changed from a ductile fracture to an intergranular fracture.

(4) During the slip process, the shear of κ carbide caused the softening of slip surface, and the increase of κ carbide size and volume fraction causes the decrease of slip band density, which leads to the decrease of the strain hardening rate with the increase of aging temperature.

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