TEM observation and strengthening mechanism of cementite nanoparticles of heterogeneous structure 1045 steel

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Abstract

A heterogeneous structural (HS) 1045 steel prepared by aluminothermic reaction (AR) casting method was fabricated by combining severe deformation cold rolled with subsequently heating. Its detailed microstructure evolution, mechanical properties, strengthening and toughening mechanisms were investigated. The heterogeneous structure (HS) is characterized with trimodal grain size distribution of ferrite (with microcrystalline grain sizes of >1000 nm, ultrafine grain sizes of ∼100 nm, nanocrystalline grain sizes of ∼100 nm grains, respectively) and cementite nanoparticles (with particle sizes of 11∼120 nm). The HS 1045 steel shows an outstanding strength–ductility synergy. When the cold rolled steel was heated at 400°C with 1 h, comparing with casting steel, the yield strength and tensile strength increased by 87.4% and 35%, to 1420 and 1602 MPa, respectively, and surprisingly, the ductility was only with a small sacrifice, the elongation rate is maintained at the level of 10.6%. The improved strength is mainly attributed to multiple strengthening mechanisms, fine-grain strengthening, cementite nanoparticles strengthening, dislocation strengthening, and solution strengthening. The reasonable ductility can be attributed to the heterogeneous structure, as it can offer an extra work hardening ability introduced by generation of geometrically necessary dislocations (GNDs) near heterointerfaces.

1. Introduction

1045 carbon steel consisting of ferrite and lamellar pearlite is widely used in the field of industrial production and manufacturing because of its excellent formability and low cost. However, its relatively low strength has been unable to meet the requirements of modern industry [1]. Although the steels with high strengths have been produced by increasing alloy contents and changing manufacturing processes, but it often leads to a significant increase in costs and notable degradation about weldability. In recent years, the strength of 1045 steel was significantly improved through refining ferrite grain size [2, 3]. The research of Park et al [4] and Wang Guodong et al [5] found that the strength of the plain carbon steel could be effectively improved when grain size less than 2 μm. However, the yield strength ratio was close to 1, and the working hardening ability was completely lost. Therefore, the contradiction of strength and ductility of nanocrystalline/ultrafine grained (UFG) steel material limits its wide applications in more critical areas. In order to resolve this issue, many advanced nanostructure design strategies have been explored to overcome this contradiction, including hierarchical nanotwinned (HNT) structures, gradient structures, bimodal grain distribution, and heterogenetic nanostructures [6–8]. In addition, many studies indicate that dispersed second phase particles can improve the work-hardening capability of nanocrystalline/UFG ferrite matrix, leading to a better ductility [9, 10].

The pearlite composed of alternating layers of ferrite and lamellar cementite. As a brittle phase, the hard lamellar cementite phase provides strength for 1045 steel, but it also leads to the deterioration of ductility. In
recent years, many studies found that when the lamellar cementite was granulated to nanometer size, and dispersed into an UFG ferrite matrix, the carbon steel can achieve a good combination of high strength and applicable ductility [11–13]. The lamellar cementite was found to decompose after further cold drawing, and dense cementite nanoparticles were precipitated when heating at 200 °C and 300 °C for 60 min [14]. Both strength and ductility of cold drawing wires were found to be obtained simultaneously. The increase of strength is believed to result from precipitation hardening of the cementite nanoparticles. The enhanced ductility may originate from the improvement in work hardening capacity through the combination of hard/soft phase. However, its plasticity still can not meet the actual production needs, and the application fields of steel wire is very limited [11–14].

In this work, we consider applying the strengthening theory of cementite nanoparticles to block 1045 steel. The 1045 carbon steel samples with nanolamellar pearlite were prepared by aluminothermic reaction (AR) casting. Because of a large super cooling degree and clean melt was provided in solidification process, the 1045 steel prepared by aluminothermic reaction casting has small grain size and pearlite lamellar spacing [15]. Then, the steel was rolled with a severe deformation rolling at room temperature and subsequently heated at a lower temperature. A heterogeneous structure consists of ‘hard’ nano-cementite particles/UFG ferrite and ‘soft’ microcrystalline ferrite was fabricated. The design of this structure is expected to achieve both high strength and high ductility of 1045 steel. The transmission electron microscopy (TEM) and x-ray diffraction (XRD) analysis were presented with the aim to reveal the mechanism about the lamellar cementite dissolution and cementite nanoparticles precipitation. In addition, the tailoring mechanism of mechanical properties by controlling the shape and size of the cementite were also investigated. We hope that this structural design should be applicable to
the majority of steel materials and can be implemented in any steel fabrication without more modification about existing facilities.

2. Experimental section

The 1045 carbon steel was prepared by AR casting, refer to previous studies [15–17], and the flowchart of specific preparation process is shown in figure 1, the schematic diagram of preparation equipment is shown as figure 2. The designed chemical composition is shown in table 1. The reaction material ratio (table 2) were calculated according to stoichiometry of the aluminothermic reaction (1). In order to reduce the adiabatic temperature of the reaction system, extra 25 wt% iron powder was added as the heat absorbent.

\[ \text{Fe}_2\text{O}_3 + 2\text{Al} = \text{Al}_2\text{O}_3 + 2\text{Fe} \]  

After casting, steel blocks, of size approximately Φ120 × 15 mm, were cut into 80 × 40 × 5 mm for cold rolling. The samples were rolled at room temperature with an accumulative thickness reduction about 85%. Then, the above plates were heated at annealed at 300, 400, and 500 °C with 1 h, respectively, and subsequently furnace cooled to room temperature. All samples were processed along the rolling direction for microstructure characterization observation.

XRD (D/Max-2400 with Cu Kα radiation) analysis was used to determine the phase composition of 1045 steel. The distribution of residual aluminum from thermite reaction was analyzed using field emission electron probe microanalysis (EPMA-1600). The electron back scattering diffraction (EBSD, Quanta450FEG) analysis was conducted to accurately investigate the variations of the ferrite grain size, the EBSD samples previously electropolished with perchloric acid electrolyte (10 Vol%) at a voltage of 20 V and a current of 0.50 mA after mechanical polishing. The obtained data was analyzed using Channel 5 software of HKL company. The precipitation and growth of cementite nanoparticles was characterized by TEM. For the observation in a JEM-2010 TEM at 200 kV, thin foils with Φ3 mm × 50 μm were obtained from sheet specimen though sanding, and then twin-jet electro-polished (an electrolyte of 2% perchloric acid in ethanol) at the temperature of
−20 °C~−15 °C, finally, ion-cleaned with 30 min (front and back sides) in a low angle using precision ion polishing system (Gatan 695.C) to remove the oxidation layer. As shown in figure 3, the treated 1045 sheets were cut along the rolling direction into dog bone-shaped specimens, with final thickness of 0.75 mm. Uniaxial tensile
Figure 7. Bright field TEM image (a), SAED image (b), dark field TEM image (c), size distribution of cementite nanoparticles (d) of the rolled steel strips after heating at 300°C with 1 h.

Figure 8. Bright field TEM image (a), SAED image (b), dark field TEM image (c), size distribution of cementite nanoparticles (d) of the rolled steel strips after heating at 400°C with 1 h.
tests were performed on a Shimadzu AT10 t machine with a crossing speed of 0.2 mm min⁻¹ at room temperature. All tests were operated three times to guarantee the data consistency. A field emission scanning electron microscope (SEM, JSM-6700F) was used to characterize the appearance of fracture.

3. Results

Figure 4 show the XRD patterns of the rolled steel strips before and after heating at different temperatures. The XRD patterns of the rolled steel belong to (110), (200), (211) and (220) ferrite peaks, and no other diffraction peaks appeared after heating at 300 °C with 1 h. When the heating temperature increased to 400 °C, the (201), (411) and (204) cementite peak can be clearly observed, it means that dense cementite nanoparticles were precipitated. After heating at 500 °C with 1 h, the cementite peak becomes inconspicuous due to the agglomeration of cementite nanoparticles. In addition, as the increase of the heating temperature, the diffraction peak intensity of (200) and (211) was obviously enhanced, it shows that the texture of deformed ferrite crystal changed after heating at different temperatures, and it will be further analyzed in the EBSD results below.

The element distributions of the casting 1045 steel are given in figure 5. According to the EPMA results, the material matrix is composed of Fe element. The carbon-rich region is pearlite phase, and the remaining region is the ferrite phase. In addition, due to the incomplete reaction of Al element in the thermite reaction process, a part of Al element remained in the material.

Figures 6–9 show the TEM bright field image, corresponding selected area electron diffraction (SAED) pattern and the dark field image corresponding to the diffraction ring segment (white circle) in the SAED pattern, the cementite nanoparticles size distribution of the rolled steel strips before and after heating at different temperature. As shown in figure 6(a), the pearlite structure consists of black cementite and gray ferrite matrix, the cementite thickness is obviously thinner after cold rolling with 85% thickness reduction, the average thickness is only about 16 nm, some cementite lamellae become obviously bent after a large deformation, and the lamellar cementites are crushed to be nanoparticles whose orientation is perpendicular to the rolling direction. According to the calibration of SAED in figure 6(b), the bright particles in the dark field image are cementite nanoparticles, through calculating ten different dark field images using IPWIN6 software, the mean size of cementite nanoparticles is about 11 nm (figure 6(d)). When the rolled 1045 steel was heating at 300 °C with 1 h, the thin cementite sheets are still bent and entangled; dense cementite nanoparticles are precipitated.
compared to the rolled state, and more evenly distributed in the matrix of steels (figure 7(c)), through calculating ten different dark field image; the mean size of cementite nanoparticles is about 15.29 nm (figure 7(d)). When heating temperature raise to 400 °C, the lamellar cementite is not observed in the bright field image, and dense cementite nanoparticles can be clearly observed in the dark field image (figure 8(c)). Compared with heating at 300 °C, the number and size of cementite nanoparticles increased significantly. The statistical results show that the mean size of cementite nanoparticles is increased to 23 nm, some size larger than 100 nm of cementite nanoparticle are caused by the crushing of lamellar cementite during rolling process (figure 8(d)). As the heating temperature continues to raise to 500 °C, there are no lamellar cementite in the bright field image. After calculating ten different dark field image (figure 9(d)), the number of cementite nanoparticle decreased significantly because of agglomeration and growth of nanoparticles, and the mean size of cementite nanoparticles is increased to 41 nm, even the largest cementite nanoparticle size is up to about 120 nm.

The EBSD orientation maps were carried out for the determination of the ferrite grain size of the rolled steel strips before and after heating at different temperature, as shown in figures 10(a)–(c). Accordingly, mean grain sizes of 897, 703 and 848 nm were acquired at cold rolling with 85% thickness reduction and then heating at 400 °C and 500 °C for 1 h, respectively, as shown in figure 10(d). The quantitative proportion of UFG grains was 72.9%, 81%, and 73.7%, respectively. The quantitative proportion of grains with size smaller than 250 nm is 37%, 46% and 37.9%, respectively [16]. The inverse pole figure (RD is rolling direction, TD is transverse direction, ND is normal direction) also shows that the steel exhibited strong texture. As shown in figure 11, after rolling with 85% thickness reduction, the texture of {111}〈110〉 is formed, and when the rolled steel heating at 400 °C and 500 °C for 1 h, the texture is {112}〈110〉. In addition, the channel 5 software was used to analyze the EBSD results. As show in figure 12, after heating, the number of recrystallization grains of rolled steel with 85% thickness reduction increased significantly. It was 6.5% and 7.7% respectively after heating at 400 °C and 500 °C for 1 h, and the corresponding number of substructure and deformed grains decreased.

The true stress-true strain curves of the rolled 1045 steel strips before and after heating at different temperatures are shown in figure 13. The yield strength ($\sigma_{0.2}$), ultimate tensile strength ($\sigma_b$), and elongation ($\delta$), taken from the true stress-true strain are summarized in table 3. Figure 12 depicts the fractographs of the rolled steel and the rolled steel strips after heating at different temperatures, respectively. These figures show that all

Figure 10. EBSD micrographs of the rolled samples (a) before and after heating at 400 °C (b), and 500 °C (c) with 1 h and statistical distribution of ferrite grain size (d). Reprinted by permission from Springer Nature Customer Service Centre GmbH: Springer Nature Metals and Materials International [16].
1045 steels mainly consist of dimples, microvoids, and also some cleavage facets are observed on the fracture surfaces. The cementite nanoparticles provided the nucleus for the formation of dimple, and the SEM pictures of the fracture surfaces proved that the main mechanisms of voids nucleation in 1045 steels are lamellar pearlite cracking and ferrite-lamellar pearlite interface decohesion. The different tensile properties of ferrite and lamellar pearlite lead to decohesion at their interface. The plasticity of lamellar pearlite is less than the ferrite result in the strain incompatibility and initiation of voids at the interface. According to the number and size of microvoids and dimples, the steels exhibit different ductility. As shown in figure 14(a), lamellar cementite were broken into granules; the mean size of dimples is 42 nm. After heating at 300 °C with 1 h, the number of microvoids and dimples was increased, distinctly; and the mean size was increased, through calculating five different fracture pictures, the mean sizes of small dimples and large dimples are 20 nm and 32 nm respectively (figure 14(b)). When the heating temperature increased to 400 °C, the same as the variation trend of cementite nanoparticles, more cementite nanoparticles are precipitated. The mean size of dimples was increased than that of 300 °C, drastically. Figure 14(c) shows the mixed fracture mode of quasi-cleavage and ductile fracture. With increase of the heating temperature to 500 °C, the number and mean size of dimples increased greatly, and the mean size of dimples is 48 nm because of agglomeration and growth of nanoparticles (figure 14(d)).
Figure 12. Distribution and volume fraction of recrystallized grains, substructures and deformed grains of the rolled samples (a) before and after heating at 400 °C (b), and 500 °C (c) with 1 h and the histogram of statistical result (d).

Figure 13. Tensile true stress-true strain curves of the rolled strips before and after heating at different temperatures with 1 h.

Table 3. Tensile properties of the rolled strips before and after heating at different temperatures.

| Condition | $\sigma_b$ (MPa) | $\sigma_{0.2}$ (MPa) | $\delta$ (%) |
|-----------|-----------------|---------------------|-------------|
| CR-85%    | 1232 ± 5        | 1212 ± 5            | 3.1 ± 0.5   |
| 300 °C-1 h| 1411 ± 8        | 1337 ± 7            | 2.1 ± 0.8   |
| 400 °C-1 h| 1602 ± 6        | 1420 ± 5            | 10.6 ± 1.2  |
| 500 °C-1 h| 1289 ± 4        | 1013 ± 6            | 17.4 ± 1.8  |
4. Discussions

A structure of microcrystalline/UFG ferrite + nanolaminate pearlite was fabricated by AR casting method [15–17]. According to EPMA results, there is residual Al element in 1045 steel matrix, and the effects of Al on the microstructure of casting 1045 steel are mainly as follows: firstly, Al element can increase the temperature of \( A_1 \) from 730 to 760 °C, it led a greater degree of undercooling during solidification; secondly, the addition of Al element hinders the coarsening of cementite lamellae, and the cementite lamellar thickness and ferrite grain size are refined [18].

When the casting 1045 steel sustained multi-pass rolled at room temperature, with the increase of thickness reduction, the microstructure of ferrite phase and cementite phase changed obviously. For ferrite microstructures, during the cold rolling, the dislocation density increased gradually, and the dislocations were accumulated and tangled with each other, which provided the conditions for subgrain nucleation. After cold rolling with 85% cumulative deformation, dislocations near the subgrain boundaries were rearranged or annihilated, finally, it resulted in a refinement of ferrite grain size. For pearlite microstructures, the spacing of the cementite lamellae was refined, drastically, and part of the lamellar cementite was broken into granules [19–21]. Such a microstructure evolution can be observed by SEM and TEM. There are two main reasons for refinement of the cementite lamellae. (1) deformation of cementite after suffering severe cold rolling; (2) decomposition of cementite. Additionally, heavy cold rolling gave rise to a stress concentration at the interface between ferrite and cementite. In order to accommodate increasing deformation and release the stress build-up, the lamellar cementite was broken. Moreover, the decomposition of cementite was always accompanied by the formation of supersaturated ferrite. Based on the results and relevant interpretations of previous studies [14, 21], a high dislocations density in the interfaces between ferrite and cementite was introduced after severe deformation cold rolling. Then, the binding energy of interaction of carbon atoms with dislocations is higher.
than that with iron atoms in cementite, which resulted in the carbon atoms segregate in the ferrite cell/grain boundaries. Finally, through the diffusion of carbon atoms, the oversaturated ferrite was formed with increase of the dislocation density.

With the increase of cold rolling deformation, the number of dislocations, subgrain boundary, and lattice distortion increased significantly, which provided more channels for carbon atoms diffusion. Based on the experimental results, dense cementite nanoparticles precipitated when the rolled steel heated at a lower temperature. There are two mechanism responsible for it. On one hand, deformation energy storage in cementite lamella went up significantly, and metastable cementite was more easy to dissolve and spheroidize. Another hand, during low temperature heating, carbon atoms diffuse from the supersaturated ferrite, and then combined with iron atoms to form cementite nanoparticles. For the cold rolled ferrite area, as the heating temperature is lower, there was no significant recrystallization after heating; only part of subgrain coalesced and grew, and the ferrite grain size decreased slightly [19–21].

According to the results about the relationship between microstructure and strength, there are four strengthening mechanisms contributing for the steel strength: fine-grain strengthening related to ferrite grain size, second phase strengthening related to cementite thickness and cementite nanoparticle size; dislocation strengthening related to the dislocation density in the ferritic phase region; solution strengthening related to the carbon concentration in the ferrite phase. And by analyzing the microstructure evolution of rolled steel after heating, the key contributor to the ultrahigh strength observed in the HS 1045 steel is precipitation strengthening of cementite nanoparticles. Surprisingly, the HS 1045 steel achieved an excellent synergy of high strength and applicable ductility. It is mainly attributed to the heterogeneous deformation-induced (HDI) strain hardening. The elastic modulus across two heterointerfaces (ferrite/nanoparticles, microcrystalline ferrite and UFG ferrite) appeared change gradually [7–10]. Upon applied straining, the large strain gradient existed across the heterointerfaces due to plastic incompatibility. Thus, dense GNDs are generated near heterointerfaces to accommodate deformation (figure 15). The capacity dislocation of accumulation of the heterogeneous structure was improved drastically. Furthermore, the interfaces of heterogeneous structure enhanced the ability to resist damage and acquired a greater tolerance to interfacial strains through layer separation [4, 22].

The fabricated HS 1045 steel exhibited an excellent synergy of strength and ductility. S Ramtani et al. [23] treated it as composite materials. During tensile deformation, the soft domains will be stretched first, and plastic deformation started after reaching the yield strength. When the stress increases to reach the yield strength of nanocrystals, cracks or holes will initiate at the heterointerfaces and hard domains. The stress continues to increase, the crack will propagate forward until reach soft domains. It will be suppressed, and the crack tip will be passivated and bridged. Crack broadening and propagation encounter a tough barrier, which effectively prevents the overall fracture of the material. When stress increases to each crack interconnect with each other, and the soft domains was also unable to restrain, the overall fracture of heterogeneous structure 1045 steel
occurred. This is the key of HS 1045 carbon steel to acquire an outstanding synergy of superior strength and ductility [22–25].

5. Conclusion

In conclusion, severe deformation accumulative cold roll bonding in conjunction with subsequent heating at lower temperatures offer an exciting strategy for producing a bulk HS 1045 steel. The heterogeneous structure of the 1045 steel is characterized with UFG ferrite and cementite nanoparticles embed in coarse-grained ferrite matrix, which has never been observed in carbon steel to date. It shows an ultrahigh yield strength (ultrahigh yield strength of 1.4 GPa and ultimate strength of 1.6 GPa) compared with corresponding coarse-grain steel, and also overcomes strength–ductility contradiction compared with UFG ones. Except for to grain refinement strengthening, the heterogeneous structure can introduce an extra strengthening effect. Both the bimodal grain size distribution of ferrite and the cementite nanoparticles increase the work hardening ability and thus help with retaining ductility. The generation of GNDs near heterointerfaces reduced mechanical incompatibility of heterogeneous structure. Back stress as an extra strain hardening is believed responsible for the strengthening in HS 1045 carbon steel. The structural designs reported here open a door for large-scale manufacturing of nanostructures carbon steels with excellent combinations of strength and ductility.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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