Effect of Annealing Temperature on Properties of Cold-Rolled Fe-Mn-Al-C Low-Density Steel: Postprint

Authors: Yang Fuqiang, Song Renbo, Li Yaping, Sun Ting, Kaikun Wang, well-being

Date: 2023-03-18T00:00:00+00:00

Abstract

The mechanical properties, metallographic microstructure, XRD patterns, and fracture behavior of cold-rolled Fe-Mn-Al-C steel were systematically analyzed within the temperature range of 850–1050℃ to investigate the transformation mechanisms of austenite, ferrite, carbides, and mechanical properties during annealing. The results indicate that after annealing at 850℃, the microstructure of cold-rolled Fe-Mn-Al-C steel comprises austenite + banded δ-ferrite + α-ferrite + -carbide; the intergranular network ferrite and high carbide content confer high strength but extremely poor ductility to the steel sheet, resulting in cleavage fracture. At 900–1050℃, the steel matrix is austenitic, the α-ferrite content decreases, and the banded δ-ferrite fragments into discontinuous island-like distributions. When the growth of δ-ferrite exceeds that of the austenite structure, the ferrite content increases and XRD peak intensities rise. The transformation of the annealed microstructure leads to a decrease in tensile strength with increasing temperature, while the elongation after fracture improves. At 1000℃, the product of strength and ductility reaches its maximum value, achieving an optimal combination of strength and toughness, with a tensile strength of 1003.1 MPa, elongation after fracture of 41.28%, and a product of strength and ductility of 41.41 GPa · %. To obtain good strength-toughness properties in cold-rolled Fe-Mn-Al-C steel, the annealing temperature must not be lower than 950℃. Furthermore, the measured density of Fe-Mn-Al-C steel is 6.55 g · cm^{-3}, demonstrating a significant weight reduction of 16.6%.

Full Text

Preamble

Vol. 29 No. 2
CHINESE JOURNAL OF MATERIALS RESEARCH
February 2015

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YANG Fuqiang, SONG Renbo*, LI Yaping, SUN Ting, WANG Kaikun, KANG Tai
(School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing 100083, China)

ABSTRACT
The mechanical properties, metallographic microstructure, XRD patterns, and fracture behavior of cold-rolled Fe-Mn-Al-C steel were systematically analyzed within the temperature range of 850-1050℃ to investigate the transformation 规律 of austenite, ferrite, carbides, and mechanical properties during annealing. The results show that after annealing at 850℃, the microstructure of the cold-rolled Fe-Mn-Al-C steel consisted of austenite + banded d-ferrite + a-ferrite + -carbides. The intercrystalline network ferrite and high carbide content endowed the steel with high strength but extremely poor plasticity, resulting in cleavage fracture. At 900-1050℃, the steel matrix became austenitic, with decreasing a-ferrite content and banded d-ferrite fragmenting into discontinuous island-like distributions. When d-ferrite grain growth exceeded that of austenite, the ferrite content increased and XRD peak intensities rose. These microstructural transformations caused tensile strength to decrease while elongation increased with rising temperature. The optimal combination of strength and ductility was achieved at 1000℃, with tensile strength of 1003.1 MPa, elongation of 41.28%, and a strength-ductility product of 41.41 GPa·%. To obtain satisfactory strength and toughness in cold-rolled Fe-Mn-Al-C steel, the annealing temperature should not be below 950℃. Additionally, the measured density of Fe-Mn-Al-C steel was 6.55 $g \cdot cm^{-3}$, demonstrating a significant weight reduction of 16.6%.

KEY WORDS metallic materials, low density steel, annealing temperature, microstructure, mechanical properties

Received August 11, 2014; in revised form December 2, 2014
Correspondent: SONG Renbo, Professor, Tel: (010)82377990, E-mail: songrb@mater.ustb.edu.cn

Introduction

To address the increasingly severe energy crisis and environmental pollution, the modern automotive industry is trending toward lightweight, energy-efficient, corrosion-resistant, and safe designs, predominantly utilizing high-strength or ultra-high-strength steel sheets to reduce thickness and achieve weight reduction. Adding substantial amounts of lightweight elements to Fe-Mn-Al-C steel can increase the lattice constant and reduce density due to their lower atomic mass while maintaining excellent strength and toughness. This steel exhibits a favorable strength-ductility combination similar to transformation-induced plasticity (TRIP) and twinning-induced plasticity (TWIP) steels, along with good corrosion resistance. First investigated in the 1950s as a potential replacement for Cr-Ni stainless steel, its combination of high strength, toughness, and low density makes it attractive for automotive lightweighting. In recent years, research institutions worldwide have devoted significant effort to developing low-density, high-strength automotive steels, particularly the Max Planck Institute for Iron Research in Germany and Hanbat National University in Korea, which have developed fully austenitic, austenite-based, and ferrite-based low-density automotive steels.

Aluminum addition significantly increases the austenite stacking fault energy and suppresses -martensite transformation. High-manganese steels with high Al content can obtain austenite + ferrite dual-phase structures. Unlike fully austenitic structures, dual-phase Fe-Mn-Al-C steels offer lower density, higher strength, and more pronounced weight reduction effects. Considering automotive industry needs and weight reduction benefits, dual-phase Fe-Mn-Al-C steel will become a key research focus. As a second phase in steel, ferrite benefits the initial work hardening rate and tensile strength, but its content and distribution affect plasticity and fracture behavior. Therefore, it is necessary to investigate the effect of annealing temperature on microstructure and mechanical properties to determine the transformation 规律 of the dual-phase structure, carbide precipitation, and optimal processing parameters.

Experimental Materials and Methods

The experimental material was low-density, high-strength, high-toughness Fe-Mn-Al-C steel. A 20 kg ingot was cast using a vacuum melting furnace, with main chemical compositions listed in Table 1 . The ingot was forged into a square billet with cross-section dimensions of 40 mm × 80 mm for hot rolling experiments. After homogenization at 1170℃ for 2 h in a heating furnace, the billet underwent six-pass hot rolling with a starting temperature of 1050℃, finishing temperature of 850℃, and coiling temperature of 550℃, resulting in a final thickness of 4.8 mm. Following water quenching after holding at 1050℃ for 1 h, cold rolling was performed using a four-high mill to a final thickness of 1.36 mm, corresponding to a cold reduction of 71.7%. The annealing process consisted of holding at 850-1050℃ for 30 min followed by water quenching.

Non-proportional standard tensile specimens with a gauge length of 50 mm were cut along the rolling direction from both cold-rolled and annealed sheets. Mechanical properties were measured using a CMT4105 electronic universal testing machine at a strain rate of 10^{-3} s^{-1}. Hardness was measured using a 430SVD digital Vickers hardness tester (30 kg load). Phase constituents of specimens annealed at different temperatures were analyzed using a DMAX-RB rotating anode X-ray diffractometer (XRD, Cu target) operating at 40 kV and 150 mA. Metallographic specimens were mechanically ground and polished, etched with 4% nitric acid alcohol solution, and examined using a ZEISS EVO 18 scanning electron microscope (SEM) for microstructural observation and energy-dispersive spectroscopy. The steel density was measured using a Sartorius BSA2245 electronic analytical balance as 6.55 $g \cdot cm^{-3}$, representing a 16.6% reduction compared to pure iron.

2.1 Mechanical Properties

Room-temperature tensile tests revealed that the cold-rolled Fe-Mn-Al-C steel had a tensile strength of 1744.4 MPa, yield strength of 1553.2 MPa, elongation of 4.00%, and a strength-ductility product of 6.98 GPa·%. The cold-rolled sheet exhibited high strength but poor plasticity, necessitating annealing treatment at 850-1050℃ for 30 min to improve microstructure and enhance strength-toughness balance. The mechanical properties of steel sheets after annealing at different temperatures are listed in Table 2 . The specimen annealed at 850℃ fractured during tensile testing, preventing measurement of tensile and yield strengths. Its hardness was measured at 400.7 HV, corresponding to an estimated tensile strength of approximately 1300 MPa based on the strength-hardness conversion relationship, with an elongation of only 0.72%. This temperature produced high strength but extremely poor plasticity.

The variation of mechanical properties with temperature is illustrated in Figure 1 [Figure 1: see original paper]. Both tensile strength and hardness gradually decreased with increasing temperature from 850℃ to 1050℃, while elongation continuously increased. The strength-ductility product, an important indicator of comprehensive toughness, reached its maximum value of 41.41 GPa·% at 1000℃. At 1050℃, although plasticity improved further, tensile strength decreased to 953.7 MPa, causing the strength-ductility product to decline slightly to 41.20 GPa·%. Compared with the cold-rolled sheet, annealing at 900-1050℃ significantly improved plasticity and strength-toughness balance.

Figure 2 [Figure 2: see original paper] presents the true stress-strain curves and work hardening curves for specimens annealed at 900-1050℃. The steel exhibited continuous yielding without an obvious yield plateau. True stress increased approximately linearly with true strain, with peak stress decreasing as annealing temperature increased. The work hardening curves (q = $d\sigma/d$) derived from the true stress-strain curves are shown in Figure 2b. The specimen annealed at 900℃ showed high initial work hardening that decreased rapidly with strain, resulting in lower elongation. In contrast, specimens annealed at 950-1050℃ displayed pronounced continuous work hardening behavior. The work hardening rate showed some fluctuation at strains below 0.10, then gradually decreased at strains above 0.10, reaching zero at peak stress. This continuous work hardening caused true stress to increase with true strain, demonstrating an excellent combination of high strength and high plasticity.

2.2 Microstructure Evolution

Annealing temperature significantly affected the phase composition of Fe-Mn-Al-C steel. Figure 3a [Figure 3: see original paper] shows the SEM micrograph after annealing at 850℃, revealing that some ferrite retained its banded morphology distributed along the rolling direction as high-temperature d-ferrite, while a-ferrite precipitated at austenite grain boundaries forming a network structure. At 900℃, banded d-ferrite began to fragment and decompose, a-ferrite content decreased, and prominent twin structures appeared within austenite grains (Figure 3b). At 950℃, carbides completely dissolved, with obvious grain boundary migration and grain growth observed (Figure 3c). At 1000-1050℃, a-ferrite continuously decreased, while d-ferrite progressively fragmented into island-like structures distributed discontinuously in the austenite matrix. Both d-ferrite and austenite grains gradually coarsened with increasing temperature, with annealing twins traversing austenite grains.

XRD analysis of specimens annealed at 850-1050℃ revealed that the 850℃ annealed specimen primarily consisted of austenite + ferrite + -carbides. The ferrite peak represented the superposition of a-ferrite and d-ferrite peaks. Comparing the two-phase structure, ferrite peaks were significantly higher than austenite peaks because a eutectoid reaction occurred during annealing: $\gamma \rightarrow \alpha +$. The -carbide [(Fe, Mn)$_3$AlC] has a face-centered cubic structure with C atoms occupying the center position, making its diffraction peaks very close to those of austenite.

Quantitative phase fraction analysis was performed using the "adiabatic method" and XRD spectra. The calculation formula uses W as the mass fraction of the calculated phase, I as the cumulative peak height of the calculated phase in XRD, I and I as cumulative peak intensities of other phases, and K as the RIR ratio from the PDF card. At 950-1050℃, the cumulative peak intensity of -carbides was zero. The calculated mass fractions of each phase are shown in Figure 5 [Figure 5: see original paper]. At 850℃, ferrite reached its maximum volume fraction of 52.5%, -carbide mass fraction was 32.5%, and untransformed austenite was only 15.0%. At 900℃, the microstructure became primarily austenite matrix + ferrite + -carbides, with austenite mass fraction of 58.2% and -carbide mass fraction decreasing to 10%. With further temperature increase, carbides completely dissolved into the matrix, and a-ferrite volume fraction decreased with temperature, reaching its minimum mass fraction of 17.1% at 1000℃. At 1050℃, d-ferrite underwent no phase transformation, but grain growth exceeded that of austenite, increasing its mass fraction to 26.6% and raising XRD peak intensities.

2.3 Fracture Behavior

Figure 6 [Figure 6: see original paper] shows the tensile fracture morphologies of steel sheets under different conditions. The cold-rolled sheet before annealing exhibited dimple morphology (Figure 6a), but the dimples were shallow with sizes around 2 m. Although the cold-rolled sheet had high strength, its plasticity was poor. After annealing at 850℃, the fracture showed obvious brittle cleavage fracture with crystalline fracture characteristics. This resulted from the $\gamma \rightarrow \alpha +$ transformation at this temperature, forming network ferrite with substantial -carbide precipitation at austenite grain boundaries. These network structures and brittle carbides directly bore the load during deformation, easily fracturing to form cracks that propagated along grain boundaries, causing intergranular fracture. Additionally, the banded d-ferrite structure in the annealed microstructure exhibited poor plastic coordination, readily generating microcracks parallel to the fracture direction (Figure 6b). After annealing at 1050℃, the experimental steel demonstrated excellent strength and toughness, with the tensile fracture showing dimples of varying sizes with smooth walls (Figure 6c). This occurred because the annealed austenite matrix possessed good deformation capability and coordination, while banded d-ferrite completely fragmented into discontinuous island-like distributions, facilitating the formation of large dimples.

2.4 Comprehensive Analysis

For dual-phase Fe-Mn-Al-C steel, ferrite presence is primarily determined by high ferrite-forming element (Al) content and processing temperature. When Al content exceeds 9.5% or processing temperature exceeds the high-temperature ferrite formation temperature, a certain amount of high-temperature ferrite structure forms, which cannot be completely eliminated during subsequent processing and heat treatment. Meanwhile, a-ferrite typically appears during low-temperature heat treatment or deformation processes. However, domestic and international scholars often use the "α" symbol to represent high-temperature ferrite in dual-phase Fe-Mn-Al-C steel research. This paper distinguishes low-temperature ferrite from high-temperature ferrite based on microstructural transformations during different temperature annealing to facilitate subsequent research.

The hot-rolled Fe-Mn-Al-C experimental steel after solution treatment at 1050℃ consisted of stable austenite + d-ferrite. Due to its high stacking fault energy, the steel retained an austenite/ferrite dual-phase structure after cold rolling without martensitic transformation, while d-ferrite formed banded structures distributed along the rolling direction. Figure 7 [Figure 7: see original paper] presents the equilibrium phase diagram calculated using Thermal-Calc software and XRD measurement results. Thermodynamic calculations (solid lines) indicate the experimental steel' s melting point is 1335℃, with d-ferrite precipitating first during cooling. Below 1250℃, the structure becomes austenite + d-ferrite two-phase. The simulation results primarily reflect equilibrium microstructures, but phase mass fractions differ from XRD measurements (dashed lines) because thermodynamic calculations are based on atomic ratios without considering atomic density in each phase, whereas XRD analysis is based on peak height and mass fraction. For example, ferrite contains higher Al content but lower density, resulting in actual ferrite mass fractions lower than calculated values. Additionally, the experimental steel experienced non-equilibrium conditions through smelting, forging, hot rolling, solution treatment, and cold rolling annealing. After hot rolling and solution treatment, austenite mass fraction exceeded 80%. At 850℃ annealing, partial austenite underwent eutectoid transformation ($\gamma \rightarrow \alpha +\ $), causing austenite mass fraction to fall below calculated values while ferrite increased. At 950-1000℃, austenite mass fraction reached its maximum of 82.9%, compared to the calculated 65.1%. Meanwhile, -carbide mass fraction was 10.0% after 900℃ annealing, decreasing to 0 at 950℃, indicating the austenite eutectoid transformation temperature occurs between 900-950℃, while the calculated transformation temperature is around 850℃.

During annealing, recrystallized austenite nucleation primarily occurred at original austenite and d-ferrite interfaces. However, SEM observation revealed numerous granular structures, 1-2 m in size, distributed along the rolling direction within d-ferrite or at d/γ interfaces during 850℃ annealing transformation. The line scanning results in Figure 8 [Figure 8: see original paper] show these particles contained significantly higher Mn and lower Fe than d-ferrite. Mn segregation commonly occurs in high-manganese steels, with austenite and ferrite corresponding to Mn-rich and Mn-poor regions, respectively. The particle structures showed obvious Mn enrichment. Based on high-temperature aging behavior in Cr-Ni duplex stainless steels, secondary austenite (γ_2) formed within d-ferrite. Secondary austenite primarily forms during aging treatment, and controlling its volume fraction can improve mechanical properties.

Annealing temperature determines microstructure and mechanical behavior. At 850℃, most austenite transformed into network ferrite and carbides, leaving only about 15.0% austenite, resulting in cleavage fracture with extremely poor strength and toughness during room-temperature deformation. After 900℃ annealing, austenite transformation volume fraction decreased while maintaining an austenite matrix. High Al and C contents increased austenite stacking fault energy, with deformation occurring via dislocation slip mechanism. Although ferrite and carbides increased initial work hardening rate and tensile strength, they hindered dislocation slip in deforming austenite, increasing dislocation motion resistance and causing rapid work hardening rate decline. After 950-1050℃ annealing, reduced a-ferrite content, austenite grain coarsening, and complete carbide dissolution into the matrix caused tensile strength to decrease and plasticity to improve. To minimize the detrimental effect of -carbides on mechanical properties, annealing temperature should not be below 950℃.

Conclusions

1. Annealing temperature significantly affects the mechanical properties of Fe-Mn-Al-C steel, substantially reducing strength while improving strength-toughness balance. At 1000℃, tensile strength reaches 1003.1 MPa with 41.28% elongation, achieving the maximum strength-ductility product of 41.41 GPa・%.

2. Ferrite can increase the initial work hardening rate during tensile deformation, but high ferrite content causes rapid work hardening rate decline, resulting in high strength but poor plasticity. At annealing temperatures of 950-1050℃, Fe-Mn-Al-C steel exhibits continuous work hardening behavior with stable, high work hardening rates.

3. Network a-ferrite and -carbides formed after 850℃ annealing are detrimental to plastic deformation, causing brittle cleavage fracture during tensile deformation. Increasing annealing temperature reduces a-ferrite and carbide content, improving strength-toughness balance of cold-rolled sheets. Banded d-ferrite fragments and decomposes during annealing, gradually coarsening with temperature. To achieve good strength and toughness, annealing temperature should not be below 950℃.

References

1. TANG Di, MI Zhenli, CHEN Yulai, Technology and research and development of advanced automobile steel abroad, Iron and Steel, 40(6), 1(2005)

2. D. Suh, N. J. Kim, Low-density steels, Scripta Materialia, 68(6), 337(2013)

3. Z. H. Cai, H. Ding, R. D. K. Misra, H. Kong, H. Y. Wu, Unique impact of ferrite in influencing austenite stability and deformation behavior in a hot-rolled Fe-Mn-Al-C steel, Materials Science and Engineering: A, 595, 86(2014)

4. H. Ding, Z. Tang, W. Li, M. Wang, D. Song, Microstructures and Mechanical Properties of Fe-Mn-(Al, Si) TRIP/TWIP Steels, Journal of Iron and Steel Research, International, 13(6), 66(2006)

5. G. Frommeyer, Udo Brux, Microstructure and mechanical properties of high-strength Fe-Mn-Al-C lightweight TRIPLEX steel, Steel Research Int., 77(9), 627(2006)

6. O. Grässel, L. Krüger, G. Frommeyer, L. W. Meyer, High strength Fe-Mn-(Al, Si) TRIP/TWIP steels development-properties-application, International Journal of Plasticity, 16(10), 1391(2000)

7. J. D. Yoo, S. W. Hwang, K. Park, Factors influencing the tensile behavior of a Fe-28Mn-9Al-0.8C steel, Materials Science and Engineering: A, 508(1), 234(2009)

8. A. Imandoust, A. Zarei-Hanzaki, S. Heshmati-Manesh, S. Moemeni, P. Changizian, Effects of ferrite volume fraction on the tensile deformation characteristics of dual phase twinning induced plasticity steel, Materials & Design, 53, 99(2014)
9. I. Kalashnikov, O. Acselrad, A. Shalkevich, C. Pereira L, Chemical composition optimization for austenitic steels of the Fe-Mn-Al-C system, Journal of Materials Engineering and Performance, 9(6), 597(2000)
10. A. Etienne, V. Massardier-Jourdan, S. Cazottes, X. Garat, M. Soler, I. Zuazo, X. Kleber, Ferrite effects in Fe-Mn-Al-C triplex steels, Metallurgical and Materials Transactions A, 45(1), 324(2014)
11. H. Kim, D. Suh, N. J. Kim, Fe-Al-Mn-C lightweight structural alloys: a review on the microstructures and mechanical properties, Science and Technology of Advanced Materials, 14(1), 14205(2013)
12. M. C. Haa, K. Jin-Mo, J. Lee, S. W. Hwang, K. Park, Tensile deformation of a low density Fe-27Mn-12Al-0.8C duplex steel in association with ordered phases at ambient temperature, Materials Science & Engineering A, 586, 276(2013)
13. H. Ding, Z. Q. Wu, H. Y. Li, M. L. Huang, F. R. Cao, Microstructural evolution and strain hardening behavior during plastic deformation of Fe-12Mn-8Al-0.8C steel, Materials Science and Engineering: A, 584, 150(2013)
14. K. Lee, S. Park, Y. S. Choi, S. Kim, T. Lee, K. H. Oh, H. N. Han, Dual-scale correlation of mechanical behavior in duplex low-density steel, Scripta Materialia, 69(8), 618(2013)
15. Z. Q. Wu, H. Ding, H. Y. Li, M. L. Huang, F. R. Cao, Microstructural evolution and strain hardening behavior during plastic deformation of Fe-12Mn-8Al-0.8C steel, Materials Science and Engineering: A, 584, 150(2013)
16. V. Torabinejad, A. Zarei-Hanzaki, S. Moemeni, A. Imandoust, An investigation to the microstructural evolution of Fe-29Mn-5Al dual-phase twinning-induced plasticity steel through annealing, Materials & Design, 32(10), 5015(2011)
17. C. Seo, K. H. Kwon, K. Choi, N. J. Kim, K. Kim, J. H. Kwak, S. Lee, N. J. Kim, Deformation behavior of ferrite-austenite duplex lightweight Fe-Mn-Al-C steel, Scripta Materialia, 66(8), 519(2012)
18. G. Fargas, M. Anglada, A. Mateo, Effect of the annealing temperature on the mechanical properties, formability and corrosion resistance of hot-rolled duplex stainless steel, Journal of Materials Processing Technology, 209(4), 1770(2009)

19. H. Cho, K. Lee, Effect of cold working and isothermal aging on the precipitation of sigma phase in 2205 duplex stainless steel, Materials Characterization, 75, 29(2013)
20. H. L. X. Jin, Secondary austenite morphologies in fusion zone of welded joint after postweld heat treatment with a continuous wave laser, Journal of Materials Science & Technology, 28(3), 249(2012)
21. K. Choi, C. Seo, H. Lee, S. K. Kim, J. H. Kwak, K. G. Chin, K. Park, N. J. Kim, Effect of aging on the microstructure and deformation behavior of austenite base lightweight Fe-28Mn-9Al-0.8C steel, Scripta Materialia, 63(10), 1028(2010)
22. S. S. Sohna, B. J. Leea, S. Leea, N. J. Kimb, J. H. Kwakc, Effect of annealing temperature on microstructural modification and tensile properties in 0.35C-3.5Mn-5.8Al lightweight steel, Acta Mater., 61(13), 5050(2013)
23. J. D. Yoo, S. W. Hwang, K. Park, Factors influencing the tensile behavior of a Fe-28Mn-9Al-0.8C steel, Materials Science and Engineering: A, 508(1), 234(2009)

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