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Ultrafine Grained Steels Processed by Equal Channel Angular Pressing

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Recent development of ultrafine grained (UFG) low carbon steels by using equal channel angular pressing (ECAP) and their room temperature tensile properties are reviewed, focusing on the strategies overcoming their inherent mechanical drawbacks. In addition to ferrite grain refinement, when proper post heat treatments are imposed, carbon atom dissolution from pearlitic cementite during ECAP can be utilized for microstructural modification such as uniform distribution of nano-sized cementite particles or microalloying element carbides inside UFG ferrite grains and fabrication of UFG ferrite/martensite dual phase steel. The utilization of nano-sized particles is effective on improving thermal stability of UFG low carbon ferrite/pearlite steel but less effective on improving its tensile properties. By contrast, UFG ferrite/martensite dual phase steel exhibits an excellent combination of ultrahigh strength, large uniform elongation and extensive strain hardenability.

Keywords *: microstructure; mechanical properties; ultrafine grains; low carbon steels; ECAP*

1. Introduction

In the late 1990's, Far East Asia countries, i.e. Korea, China, and Japan, initiated the nationwide research projects developing the new types of low carbon steel for the structural use to prepare the replacement of existing infrastructures and the construction of more gigantic infrastructures in the near future.[1)] The property targets required to the new structural low carbon steels are (a) ultrahigh strength, (b) very low ductile-brittle transition temperature associated with high toughness, (c) high corrosion resistance for the maintenance-free, and (d) the least compositional modification for easy end-of-life scrap recycling, etc. One of the promising approaches achieving these targets is the grain refinement since grain refinement is the only strengthening mechanism enhancing both strength and toughness among various strengthening mechanisms. For instance, in case of low carbon ferrite-pearlite steels, the refinement of the ferrite grain size from ~10 μm to ~1 μm is expected to increase the strength as much as ~400 MPa and to decrease a ductile-brittle transition temperature from -20 C to -200 C without any compositional modification.[2)] As a result, along with an emergence of nanostructured materials, extensive efforts have been devoted to develop low carbon steels having the ferrite grain size of ~1 μm or less, i.e. ultrafine grained (UFG) steels, and the related processing technologies, especially with Far East Asia countries in the center.

Microstructural refinement of steel is usually achieved by alloying and/or thermomechanical treatments accompanying various types of phase transformation. Meanwhile, recent advance of severe plastic deformation (SPD) techniques provides another efficient access for grain refinement of metals and alloys.[3)] Of the SPD techniques, equal channel angular pressing (ECAP)[4)] is the most widely used as a model technique analyzing the physical and mechanical characteristics of its products, i.e. bulk, porosity- and contamination-free UFG materials, and the effects of the SPD processing variables on the formation of an UFG structure. Accordingly, in line with the current development of new types of low carbon steel described above, it is natural to apply ECAP to fabricate UFG low carbon steels. The purpose of this article is two-fold; (a) to review the microstructures and mechanical properties of ECAPed UFG low carbon steels and (b) to explore the ways overcoming their critical mechanical drawbacks.

2. Microstructures of UFG low carbon ferrite/pearlite steels processed by ECAP

2.1 Ferrite grain refinement

When a steel consisting of coarse ferrite grains and pearlite colonies is subjected to ECAP with either route B_c or C, nearly equiaxed ferrite grains of 0.2~0.5 μm are

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formed beyond an effective strain of ~4.[5),6)] As a rule, coarse ferrite grains are refined by their subdivision resulted from operation of the slip systems typical in BCC structure.[7)] In the first pass of ECAP, a *specific* slip system belonging to the typical slip system family for BCC structure operates, resulting in the formation of deformation bands aligned along the corresponding slip direction. Then, by the second pass (except route A), the slip direction is changed in association with sample rotation between passages. Then, *all possible slip systems* of BCC structure become activated in order to accommodate further deformation which is restricted by the deformation bands formed by the prior passage. At this stage, the microstructure was dominated mainly by ultrafine equiaxed dislocation cells or subgrains. By further ECAP passages, cell or subgrain boundaries become high angled either by their rotation to achieve even shear strain distribution at all boundaries or by absorption of lattice dislocations in the form of extrinsic grain boundary dislocations.

2.2 Microstructural evolution in pearlite colonies

Pearlite has an interwoven structure of alternative layers of ferrite and cementite. Cementite is known to be brittle under the uniaxial deformation mode due to a lack of the slip systems for homogeneous deformation related to its orthorhombic structure. However, pearlitic cementite lamellae deformed by ECAP exhibit multi-necked or curled and wavy morphology, indicating that they deform plastically.[5)] These morphological characteristics are very similar to those observed in the severely cold drawn pearlitic steel wires.[8)] In case of the severely cold drawn pearlitic steel wires, plastic deformability of pearlitic cementite is explained by activation of the additional slip systems under the hydrostatic stress state imposed by drawing.[9)] Although no detailed study has been performed at present on the active slip systems of pearlitic cementite during ECAP, its plastic deformability would be explained in the similar manner in light of the morphological similarities between the two cases.

Another noticeable phenomenon is dissolution of carbon atoms from severely deformed pearlitic cementite and their concurrent diffusion into either pearlitic ferrite or adjacent ferrite matrix during ECAP, resulting in the carbon content in ferrite matrix higher than the equilibrium value.[10)-12)] The strength of pearlite primarily depends on interlamellar spacing. Accordingly, damage of cementite associated with carbon dissolution may degrade the strength of pearlite by increasing the *effective* interlamellar spacing. However, such an effect does not influence the strength of ECAPed ferrite/pearlite steels since their ultrahigh strength is primarily attributed to UFG ferrite grains. Instead, through proper heat treatments, carbon atom dissolution can be utilized (a) to enhance a thermal stability of ECAPed UFG ferrite/pearlite steel by precipitation of nano-sized cementite particles at UFG ferrite grain boundaries,[10),11)] and (b) to create UFG ferrite/martensite dual phase steel showing an excellent combination of ultrahigh strength, large uniform elongation and extensive strain hardenability.[13)]

3. Room temperature tensile properties of UFG low carbon steels processed by ECAP

In spite of their ultrahigh strength, nano or UFG materials seem to be impractical due to very low uniform elongation and strain hardenability, which are unacceptable for the structural use, associated with limited dislocation activities at the UFG grain interior.[14)] Accordingly, recent studies on the mechanical properties of nano or UFG materials are devoted to overcome these mechanical drawbacks. In this section, the ways enhancing ductility and strain hardenability of ECAPed UFG low carbon steels (not confined to ferrite/pearlite steels) with the least degradation of strength are explored. Of several suggestions regarding this issue,[15)-17)] the two are worth examining in view of easiness of application to steel processing: (a) uniform distribution of nano-sized second phase particles and (b) strain gradient plasticity originated from the composite microstructure consisting of hard martensite and soft ferrite.

3.1 As-ECAPed UFG low carbon ferrite/pearlite steel

As shown Fig. 1, the strength of as-ECAPed UFG

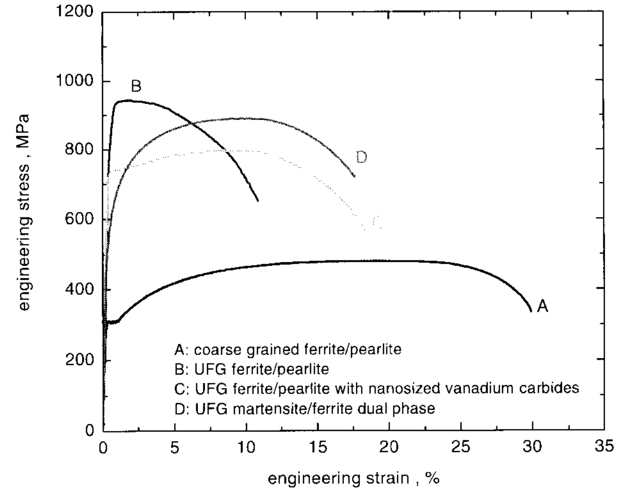

Fig. 1. Engineering stress-strain curves of UFG low carbon steels fabricated by a combined process of ECAP and various heat treatments.

ferrite/pearlite steel (curve B, ferrite grain size ≈ 0.3 μm) is over three times higher than that of coarse grained counterpart (curve A, ferrite grain size ≈ 30 μm) but the former exhibits not only very low ductility but no strain hardening. In this case, ECAP was performed up to an effective strain of ~4 with route C at 623 K. Higher ECAP strains and the use of route B_c result in only slight increase of strength and ductility.[6] A lack of strain hardenability in UFG materials including the present UFG ferrite/pearlite steel is attributed to the combined effects of (a) the UFG ferrite grain size comparable to the dislocation mean free length and (b) dynamic recovery related to the dislocation absorption rate at ferrite grain boundaries exceeding the dislocation generation rate from the source.[18),19)]

3.2 UFG low carbon ferrite/pearlite steel with uniformly distributed nano-sized vanadium carbides

The engineering stress-strain curve of ECAPed UFG ferrite/pearlite steel with uniformly distributed nano-sized vanadium carbides is shown in Fig. 1 (curve C, ferrite grain size ≈ 0.5 μm). This steel with identical composition with the steel of curves A and B except addition of 0.34 wt.% vanadium was fabricated by ECAP (identical conditions with the steel of curve B) and proper annealing for strain induced precipitation of vanadium carbides: detailed procedure was described elsewhere.[20)] Fig. 2a shows the size and distribution of vanadium carbides at the UFG ferrite grain interior, and their interaction with lattice dislocations after tensile deformation. As shown in Fig. 1, the strength of this steel reaches almost twice of that of coarse grained ferrite/pearlite steel without vanadium (curve A) but much less than that of as-ECAPed UFG ferrite/pearlite steel without vanadium (curve B). Although its ductility was improved, the strain hardenability was still too low in spite of interaction between vanadium carbides and dislocations: the Hollomon strain hardening exponent was ~0.09 and 0.25 for curves C and A, respectively.

The present authors conducted a large number of experiments by varying vanadium content, ECAP conditions, pre- and post-annealing conditions, etc in order to alter the size and distribution of vanadium carbides. As a result, it was found that the utilization of nano-sized second phase particles is rather effective on improving *thermal stability* of UFG materials but less effective on improving the tensile properties, especially strain hardenability, since their tensile properties primarily depend on the ultrafine grain size.

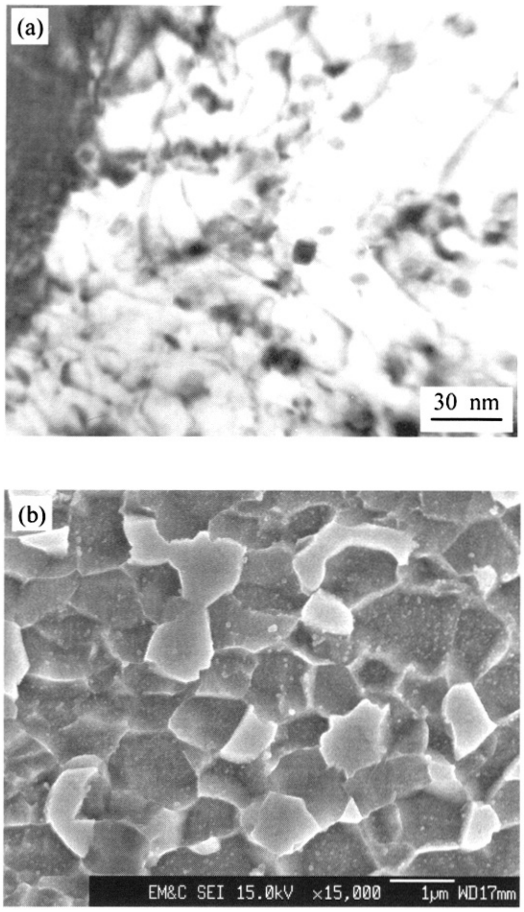

Fig. 2. (a) TEM micrograph showing the size and distribution of vanadium carbides at the UFG ferrite grain interior, and their interaction with lattice dislocations after tensile deformation of UFG low carbon ferrite/pearlite steel containing vanadium. (b) SEM micrograph of UFG ferrite/martensite dual phase steel fabricated by ECAP and subsequent intercritical annealing: bright patches are martensite.

3.3 UFG low carbon ferrite/martensite dual phase steel

In strain gradient plasticity, two kinds of dislocation play different main role on plastic deformation: statistically stored dislocations for deformation itself and geometrically necessary dislocations for strain hardening.[21)] Accordingly, an introduction of a large density of the latter would enhance strain hardenability. In case of steel, one of the representative examples explaining its tensile properties in terms of strain gradient plasticity is ferrite/martensite dual phase steel. The tensile properties of coarse grained ferrite/martensite dual phase steel is characterized by low yield strength and high ultimate tensile strength resulting in a low yield ratio, extensive rapid strain hardening from the onset of plastic deformation, relatively large uniform elongation, etc. The excellent strain hardenability of dual phase steel is attributed to the generation of fresh mobile dislocations with a high density associated with transformation accommodation in the vicinity of martensite/ferrite phase boundaries during martensite transformation[22] and these dislocations are regarded as geometrically necessary dislocations.[17]

By using ECAP of low carbon ferrite/pearlite steel and proper subsequent intercritical annealing, it is possible to fabricate UFG martensite/ferrite dual phase steel. Fig. 2b shows the uniform microstructure of UFG martensite/ferrite dual phase steel consisting of ferrite grains and isolated martensite islands of ~0.8 μm, fabricated by ECAP of an effective strain of ~4 with route C at 773 K followed by intercritical annealing at 1003 K × 10 min. This unique microstructure results from dissolution of carbon atoms from pearlitic cementite and their concurrent diffusion into UFG ferrite during ECAP, making the average carbon content in UFG ferrite to reach the equilibrium carbon content enough to form austenite during subsequent intercritical annealing.[13] The curve D in Fig. 1 shows the engineering stress-strain curve of this UFG dual phase steel. Unlike most UFG materials showing limited ductility and negligible strain hardening, this UFG dual phase steel exhibit an excellent combination of ultrahigh strength, large uniform elongation and extensive strain hardenability. The present finding demonstrates that strain gradient plasticity having the characteristic length scale independent of the grain size has high potential to store strain hardenability of nano or UFG materials.

4. Concluding remarks

In this short article, the microstructures and tensile properties of several kinds of UFG low carbon steels fabricated by ECAP and various heat treatments are reviewed. For the use of UFG low carbon steels as prominent structural materials, restoration of ductility and strain hardenability of these steels should be addressed. Nano-sized precipitates of microalloying elements such as vanadium can be utilized to improve thermal stability and to tailor the desired combination of strength and ductility, although they are not so effective to enhance strain hardenability. By contrast, UFG martensite/ferrite steel fabricated by a combined process of ECAP and intercritical annealing exhibits an excellent combination of ultrahigh strength, large uniform elongation and extensive strain hardenability in association with strain gradient plasticity.

References

1. K. Tsuzaki, *ISIJ Int.* **42**, 1325 (2002).
2. F. B. Pickering, in: Physical Metallurgy and the Design of Steels, Appl. Sci. Pub, London, UK, 1978, p.37.
3. R. Z. Valiev, R. K. Islamgaliev, and I. V. Alexandrov, *Prog. Mater. Sci.* **45**, 103 (2000).
4. V. M. Segal, *Mater. Sci. Eng.* **A197**, 157 (1995).
5. D. H. Shin, B. C. Kim, Y. S. Kim, and K. -T. *Park, Acta Mater.* **48**, 2247 (2000).
6. Y. Fukuda, K. Oh-ishi, Z. Horita , and T. G. Langdon, *Acta Mater.* **50**, 553 (2002).
7. D. H. Shin, I. Kim, J. Kim, and K. -T. Park, *Acta Mater.* **49**, 1285 (2001).
8. W. J. Nam and C. M. Bae, *Mater. Sci. Eng.* **A203**, 278 (1995).
9. J. Gil Sevillano, *Mater. Sci. Eng.* **21**, 221 (1995).
10. K. -T. Park, Y. S. Kim, and D. H. Shin, *Metall. Mater. Trans. A.* **32A**, 2373 (2001).
11. K. -T. Park and D. H. Shin, *Mater. Sci. Eng.* **A334**, 79 (2002).
12. D. H. Shin, K. -T. Park, and Y. S. Kim, *Scripta Mater.* **48**, 469 (2003).
13. K. -T. Park, S. Y. Han, B. D. Ahn, D. H. Shin, Y. K. Lee, and K. K. Um, *Scripta Mater.* **51**, 909 (2004).
14. H. Conrad, *Mater. Sci. Eng.* **A341**, 216 (2003).
15. Y. M. Wang and E. Ma, *Acta Mater.* **52**, 1699 (2004).
16. C. C. Koch, *Scripta Mater.* **49**, 657 (2003).
17. J. Aldazabal and J. Gil Sevillano, *Mater. Sci. Eng.* **A365**, 186 (2004).
18. K. -T. Park, Y. S. Kim, J. G. Lee, and D. H. Shin, *Mater. Sci. Eng.* **A293**, 165 (2000).
19. K. -T. Park and D. H. Shin, *Metall. Mater. Trans. A.* **33A**, 705 (2002).
20. K. -T. Park, S. Y. Han, D. H. Shin, Y. K. Lee, K. J. Lee, and K. S. Lee, *ISIJ Int.* **44**, 1057 (2004).
21. A. Needleman and J. Gil Sevillano, *Scripta Mater.* **48**, 109 (2003).
22. D. K. Matlock, F. Zia-Ebrahimi, G. Krauss, in: Deformation, Processing and Structure, ASM, Metals Park, USA, 1982, p.47