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Fabrication of an ultrafine-grained structure by a compositional pinning technique

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Abstract

A new metallurgical process for fabricating ultrafine-grained alloys by a compositional pinning technique is proposed. A Fe–10Mn–0.2C alloy was prepared by vacuum induction melting, and the substitutional alloying element Mn was partitioned by annealing in the austenite + ferrite region. After the partitioning, specimens were reheated to austenite temperature and cooled at varying cooling rates or cold rolled and recrystallized. Specimens quenched or slowly cooled after reheating showed ferrite-plus-austenite layered structures, while cold-rolled and recrystallized specimens showed equiaxed grain structures of ferrite plus austenite. The average grain size (or layer thickness) was <1 μm. The Mn-enriched region survived the reheat annealing and effectively acted as a barrier to ferrite growth. The quench and tempered specimens showed continuous yielding behavior, while the cold-rolled and recrystallized ones showed discontinuous yielding and serrated flow. The steels all showed excellent combinations of strength and ductility, yield strength >700 MPa and tensile strength >1100 MPa. Tensile elongation was the shortest, ~20%, in quench and tempered steel, and was ~40% in slowly cooled or cold-rolled and recrystallized steels. Possible reasons for the difference in tensile behavior are discussed. This technique opens a new way for the industrial production of various grades of ultrafine-grained alloys through a modification of the alloy composition and thermomechanical processing route.
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Keywords: Alloy partitioning; Compositional pinning; Ultrafine grain; Yield elongation; Serrated flow

1. Introduction

Grain refinement is known to be a promising method of improving the strength of alloys without impairing their ductility. In particular, grain refinement can reduce the ductile-to-brittle transition temperature (DBTT) of body-centered cubic (bcc) steels. For example, by decreasing the grain size of low-carbon steels to 1 μm, the yield strength of the steels can be increased to >700 MPa, while their DBTT can be lowered to below liquid nitrogen temperature [1,2]. Triggered by the Ultra-Steel project in Japan [3], the development of an effective way to reduce the grain size to a level of 1 μm has been one of the active research subjects in the steel industry over the last two decades. Many innovative laboratory methods of producing ultrafine-grained (UFG) steel were proposed. Various severe plastic deformation methods, including equal-channel angular pressing [4–7], accumulative roll bonding [8–10] and high-pressure torsion techniques [11–13] have been proposed. However, the production of UFG steels by these techniques was limited to laboratory scale, and the application of these techniques to massive industry-scale production is not feasible. Other techniques more oriented to industrial production have also been proposed. The

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strain-induced transformation technique is one such technique, aimed at industrial-scale production, which uses dynamic ferrite transformation during deformation between Ae_3 and Ar_3 temperatures [14–16]. The possibility of controlling the cooling rate after partial transformation of ferrite is one of the advantages of this technique, which enables control of the final microstructure for the properties. However, the reduction rate to obtain a full UFG structure by dynamic ferrite transformation is formidable, at ~70% and <800 °C, which is not achievable in present industrial hot-rolling mills. Multiple reductions in a hot-rolling mill with an extremely short inter-pass time have also been proposed [17]. In this technique, ferrite transformation occurs from the deformed austenite. The driving force for ferrite transformation would be larger, owing to the high density of dislocation, and an increase in intragranular nucleation sites is expected from deformation bands or other defect sites of austenite [18]. However, because of the high ferrite transformation temperature, the increase in nucleation rate is limited, and the grain growth cannot be suppressed, despite the fast cooling adopted after ferrite transformation.

To obtain a UFG structure, prevention of grain growth after nucleation is crucial. Techniques to minimize grain growth after ferrite formation by rolling at a lower temperature or using grain boundary pinning agents have been proposed. These techniques include heavy warm rolling of ferrite [19–21], cold rolling of martensite and subsequent short annealing [22–24] or rolling in the two-phase region [25]. During warm rolling of ferrite, dynamic recrystallization is induced [19,20] to obtain high-angle grain boundaries (HAGBs). Song et al. [26,27] also proposed that pronounced recovery annealing is more beneficial in obtaining HAGBs. Cold rolling and subsequent annealing was reported to be very effective in reducing ferrite grain size [22]. However, heavy warm rolling of ferrite for dynamic recrystallization requires very high rolling loads that modern industrial rolling mills cannot sustain, and repeated cold rolling of martensite also demands extended processing time, which limits industrial production.

Another critical issue in grain refinement, which has not yet been properly answered, is the loss of ductility observed in UFG alloys. When the grain size is reduced to the range of 1 μm, uniform elongation practically disappears, along with the loss of work hardening [2], which is unacceptable for structural applications. Localized deformation associated with the decrease in dislocation density within the grains due to dynamic recovery [28] and plastic instability [29,30] was suggested to be the reason for this loss of ductility in UFG alloys. To overcome this limitation, several techniques have been proposed. Choo et al. [31] rapidly cooled the alloy after partial ferrite transformation, to form martensite grains, and extended the tensile elongation of fine-grained low-carbon steel up to ~30%. The use of second phases to enhance work hardening of UFG steels has been explored and documented. Ohmori et al. [32] and Song et al. [27] experimented with using cementite precipitation to improve the work hardening of medium carbon steels. Duplex structures of ferrite/martensite [33–35] or ferrite/austenite [36,37] were explored. However, for full structural application of UFG steels, further work is necessary to overcome this critical shortcoming of UFG steels. Recently, Tsuji et al. [38,39] refined the grain size of twinning-induced plasticity (TWIP) steel to a sub-micron level by heavy cold working (92%) and annealing techniques. They reported tensile elongation of 50% for 0.4-μm-sized TWIP steel. TWIP steels are well known for their high work hardening rate. Self grain refinement due to twinning was attributed to the high work hardening rate of TWIP steel. Deformation twinning may also be effective in improving the work hardening of UFG steel [39].

This paper proposes a new method of reducing the grain size of ferrite-plus-austenite duplex steels by a compositional pinning technique. An average grain size of ~0.6 μm was achieved, and the tensile elongation of the alloy was ~40%. This technique can be applied to any alloy system that has a two-phase region, and the growth of grains can be suppressed by the presence of an alloy composition gradient. Various grades of steel with various strengths and ductilities may possibly be designed using this technique through the control of alloy composition, heat treatment and/or rolling schedule.

2. Experimental procedures

Fe–10Mn–0.2C alloy was prepared by vacuum induction melting. The chemical composition of the alloy is given in Table 1. Aluminum was added to prevent the precipitation of epsilon (ε)-martensite by increasing the austenite stacking fault (SF) energy [40,41], and molybdenum was added to suppress the tendency for intergranular fracture [41]. The alloy was annealed at 1100 °C for 2 h and then hot-rolled to a 4-mm-thick plate. Specimens 150 mm long and 10 mm wide were cut from the plate for subsequent heat treatments. Specimens were solution-treated at 900 °C for 20 min and then quenched in water to produce the lath martensite structure. Martensite specimens were annealed in the two-phase (ferrite + austenite) region (525 or 550 °C) for 8–32 h for Mn partitioning. During alloy partition annealing, austenite laths were produced at the martensite lath boundaries. A typical heat treatment procedure is illustrated in Fig. 1, with the schematic equilibrium phase diagram of the alloy. The Mn content in the austenite and martensite laths after alloy partitioning heat treatment were expected to be C_γ and C_α in Fig. 1, respectively. After alloy partitioning, some specimens were reheated to the austenite temperature (850 °C) and then water-quenched and tempered at 200 or 300 °C, and others

Table 1
Chemical composition of the alloy (wt.%).

C	N	P	S	Mo	Mn	Al	Fe
0.177	0.0047	0.009	0.003	0.505	10.62	2.84	Bal.

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