Role of stress-assisted martensite in the design of strong ultrafine-grained duplex steels

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Abstract—This work explains the occurrence of transformation-induced plasticity via stress-assisted martensite, when designing ultrafine-grained duplex steels. It is found that, when the austenite is reduced to a fine scale of about 300 nm, the initial deformation-induced microstructure can be dominated by parallel lamellae of ε martensite or mechanical twinning, which cannot efficiently provide nucleation sites for strain-induced martensite. Hence, α′ martensite nucleation occurs independently by a stress-assisted process that enhances transformation-induced plasticity in ultrafine-grained austenite. This metallurgical principle was validated experimentally by using a combination of transmission Kikuchi diffraction mapping, transmission electron microscopy and atom probe microscopy, and demonstrated theoretically by the thermodynamics model of stress-assisted martensite.
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1. Introduction

Strong steels that possess the combination of properties necessary for the manufacture of automobiles are the subject of intense research and development because of the increasing need for fuel efficiency, emissions reduction and safe design [1]. Nevertheless, it is recognized that there are driving forces for further alloy development in this area, and a particular focus has been on the development of variants that are strong and ductile [2–4]. Extreme grain refinement in general leads to strengthening [5,6], but the ductility is dramatically reduced [7,8]. When the boundaries are so closely spaced, the dislocations sink into the boundaries or mutually annihilate, causing the uniform ductility to become vanishingly small because of the lack of a prominent work hardening mechanism. In searching for strong but ductile ultrafine-grained steels, a source of mobile dislocation [9] or other mode of plasticity [2,4,10,11] will be required.

There are three essential plastic deformation mechanisms in austenite at ambient temperature [12]: dislocation slip, the shape deformation accompanying ε or α′ martensitic transformation and mechanical twinning. Tailoring the chemical and structural characteristics of the austenite can invoke the operation of particular modes of plastic deformation, as required. This is important when designing fine-grained steels [2,4,10,11] and those that are nanocrystalline [13,14], in order to avoid the well-known loss of ductility through necking shortly after yield. In these steels, other plastic modes, such as martensitic transformation or mechanical twinning, are essential in order to enable strain hardening. This idea has been utilized in the recent development of fine-grained duplex (austenite–ferrite or austenite–martensite) steels with 5–7 wt.% of manganese [2,4,15]. Those duplex microstructures benefit from the retained austenite, which can transform into martensite during deformation, thereby preventing plastic instabilities. However, such alloys [2,4,10,15] generally yield at stresses well below 1 GPa, but ultrahigh yield strength will also be required especially for the various intrusion applications or hole-expanded components in the automotive industry [1]. Moreover, some of the medium manganese steels require prolonged heat treatments so as to ensure solute partitioning in order to stabilize the austenite [16,17],

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making the manufacturing process both time and energy consuming.

The present work was undertaken to resolve these difficulties by designing a new alloy containing a manganese concentration of 11 wt.%. This special alloy can be very strong and ductile due to its ultrafine-grained duplex structure, produced by a short annealing. Particular attention was devoted to elucidating the metallurgical principles of the transformation-induced plasticity (TRIP) effect when the grain size is extremely small. The tensile mechanical behavior is presented together with the microstructural details which were obtained using scanning electron microscopy (SEM) based transmission Kikuchi diffraction (TKD), transmission electron microscopy (TEM) and atom probe microscopy (APM). These techniques provide direct evidence and data for the thermodynamics model, indicating an enhanced stress-assisted TRIP effect which has not previously been reported in steels of this grade.

2. Alloy design

The steel composition developed in this research is presented in Table 1. The steel was prepared by vacuum induction melting, and cast into ingots $80 \times 30 \times 12$ mm^3 in size. The alloy was designed to possess a very fine-grained austenite–ferrite duplex microstructure that could be achieved by a combination of cold rolling and intercritical annealing, i.e. heat treatment in the austenite and ferrite two-phase field. There are several novel aspects to the proposed composition. Firstly, the steel possesses a much lower carbon content than the steels reviewed in the previous section (Table 1). This ensures that cold rolling down to a sheet as thin as 1.2 mm gage thickness can be achieved from the martensitic state that predominates after hot rolling. The low carbon content also suppresses the formation of cementite, and so enables a larger temperature window for intercritical annealing. The 10–11 wt.% of manganese increases the stability of the austenite generated during intercritical annealing and yet is substantially lower than that used in the twinning-induced plasticity (TWIP) steels. The addition of vanadium provides for grain size control, and that of molybdenum for mitigating embrittlement by phosphorus and similar impurities, given the susceptibility that strong steels have to intergranular failure. Our aim was to achieve a duplex microstructure having equal volume fractions of austenite and ferrite so as to enhance the materials capacity for work hardening during deformation of the austenite.

The ingot was soaked at 1200 °C for 1 h and then hot-rolled to a 6 mm thick plate, followed by air cooling to room temperature. The plate was then reheated to 1200 °C for 30 min and then hot rolled to a 2.8 mm thick strip, with a finish rolling temperature of 850 °C. This hot-rolled strip was then air cooled to room temperature. After removing the oxide layer on the steel surface via pickling, its thickness was reduced from 2.4 to 1.2 mm via a final cold rolling step, over eight passes at room temperature.

Fig. 1a charts the equilibrium phase fractions by weight as a function of temperature, calculated using Thermo-Calc [18] with the TCFE7 database. The equilibrium weight fractions of austenite at the temperatures of 550 and 610 °C were 0.52 and 0.80, respectively. Considering the small difference in atomic density between austenite and ferrite,

the volume fractions were also close to these values. Our target austenite volume fraction of 0.5 can therefore be achieved via an intercritical anneal at either temperature. At 610 °C, it would be necessary to interrupt the heat treatment prior to equilibrium, whereas at 550 °C it would be necessary to arrest the heat treatment close to equilibrium. What is of crucial significance, however, is that these two intercritical annealing temperatures can grow austenite with significantly different characteristics. The corresponding austenite transformation kinetics are set out in Fig. 1b on the basis of dilatometry measurements made during annealing at 550 and 610 °C using the methods reported in Refs. [19,20]. The kinetic analysis of the austenite transformation reveals that approximately 100 min is required to reach the required volume fraction of austenite (V_γ) of 0.5 at 550 °C. In contrast, only 370 s is required at 610 °C. To enable the use of sufficiently large samples for tensile testing, strip samples were annealed in a box furnace. Given the relatively slow heating rate (approximately 3–4 °C s^{-1}) of this type of furnace, the intercritical annealing times were extended to 2 h and 8 min at 550 and 610 °C respectively, and this was followed by water quenching to ambient temperature. Henceforth, the samples annealed at 550 °C for 2 h are designated "550LA" and those annealed at 610 °C for 8 min are designated "610SA", where the LA and SA designations refer to the relatively long and short annealing treatments, respectively. Table 2 lists the phase ratios obtained from X-ray diffraction (XRD) analysis of both steel samples after intercritical annealing. It is seen that the final volume fractions of austenite are close to the intended V_γ of 0.5. This is a key result and, as will be seen below, highlights the success of the present alloy design, since we are able to use two different intercritical annealing temperatures to achieve the same phase constitution, yet one route is relatively rapid and the other is relatively slow. As will be demonstrated below, the fundamental nature of the austenite in each of these cases is fundamentally different.

3. Experimental procedures

XRD was performed on colloidal silica polished specimens using Cu K_α radiation. The peak shape analysis was performed using the TOPAS 3.0 software, fitted using the modified pseudo-Voigt function. The phase fractions were determined via Rietveld analysis. The dislocation density of the ferrite was evaluated using the approach detailed in Refs. [21,22].

Tensile testing was conducted at a constant strain rate of 0.001 s^{-1} using an MTS 810 mechanical testing system. The samples were machined into the ASTM E8 [23] sub-sized specimens (6 mm wide and 25 mm gauge length) along the longitudinal direction of the annealed strips. To study the deformed state, samples were extracted from the gauge regions of the tensile samples. The strain of the each sample was estimated by the local area reduction.

Samples for TEM were mechanically polished using 1200-grit sandpaper and then electropolished with an electrolyte of 10% perchloric acid and 90% acetic acid under a voltage of 15 V at a temperature of ~10 °C. The thin foils were examined in a JEOL 2100 and a JEOL 2200FS transmission electron microscope, both operating at 200 kV and well suited to the application of selected area electron