Scripta Materialia 141 (2017) 111–114

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

Regular Article

# Fabricating interstitial-free steel with simultaneous high strength and good ductility with homogeneous layer and lamella structure

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ARTICLE INFO

*Article history:*
Received 18 May 2017
Received in revised form 17 June 2017
Accepted 23 June 2017
Available online 8 August 2017

*Keywords:*
Interstitial-free steel
Heterogeneous structure
Mechanical properties
Work hardening

ABSTRACT

Annealed interstitial-free steel (IF steel) and deformed IF steel sheets were stacked alternatively into multi-layers to produce laminated IF steel through thermal-mechanical processing. After proper processing, a yield strength of 500 MPa, an ultimate tensile strength of 600 MPa (comparable to cold rolled one) and a uniform elongation around 17% can be realized. Microstructural observation by electron back-scatter diffraction revealed a characteristic hierarchical layer + heterogeneous lamella structure, namely L2 structure. The reasons for the good mechanical properties were discussed.



It is a challenge to produce metals and alloys with both high strength and good ductility. This is because a material may be either strong or ductile but rarely both at once. Recently, a series of breakthroughs have been realized by tailoring nanostructured grains to achieve decent ductility while keeping the high strength. Examples are bimodal (or multi-modal) grain size distribution in Cu and Ni [1,2], gradient nano-grained Cu, Ni and IF steel [3–7], heterogeneous lamella structured Ti [8] and laminated Cu/Cu composite [9]. It is realized that by introducing a heterogeneous structure into those metals, work hardening capability can be improved and thus higher uniform ductility can be achieved, with only a relatively small loss of strength [4].

Here, we report a new strategy for single phase IF steel with the aim to realize a superior combination of strength and ductility. The idea is to produce a heterogeneous lamella structure by introducing different initial structures namely stacking of deformed and annealed layers alternatively into multi-layers. The stacked plates were "welded" together by hot compression, followed by thermomechanical processing. It is shown that a good combination of strength and uniform tensile elongation in IF steel, together with good stability and reproducibility, was obtained by this strategy. Transmission electron microscope and electron back-scatter diffraction (EBSD) investigation were used to characterize the microstructure and the mechanical properties are discussed based on this characterization.

The initial material was 1 mm thick Ti-added commercial cold rolled (77.8% reduction in thickness) IF steel sheets. The chemical composition of the IF steel was 0.012 wt% C-0.005 wt% Si-0.1 wt% Mn-0.061 wt% Ti. One cold rolled sheet was annealed at 780 °C to complete recrystallization with an average grain size of about 20 μm. The cold rolled and the annealed sheets in the following are called CR and AR, respectively. The CR and AR steel sheets were cut into plates with a diameter of 15 mm. After surface cleaning by polishing with grinding papers and degreasing with acetone, the CR and AR steel sheets were stacked to 11 layers in an alternate sequence, as shown in Fig. 1(a) step 1. During the stacking, all plates were aligned along their original rolling direction (RD). The stacked multilayer plates were hot compressed to a thickness reduction of 40% at 500 °C by using a Gleeble 3800 thermo-mechanical simulator in vacuum to weld the layers together. The hot-compressed specimens were air cooled and then cold forged (CF) at room temperature to a thickness of about 1.6 mm by a hydraulic press, as shown in Fig. 1(a) step 2. After cold forging, the nominal deformation strains of the initial CR and AR layers are 96.8% and 85.5% corresponding to von Mises strains of 4.0 and 2.2, respectively. The interfaces between the original AR and CR layers can be recognized after hot compression, but cannot after cold forging (Fig. 1(a) step 3). However, the differences can be seen in the microstructure after etching with a 5% Nital solution. The elongated feature in the CR layers is more clear which relates to the higher nominal deformation strain in the CR layers than that in the AR layers. The cold forged specimens were then annealed at various temperatures ranging from 500 °C to 700 °C for different times. Tensile tests were performed along the original RD direction to evaluate the

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http://dx.doi.org/10.1016/j.scriptamat.2017.06.044
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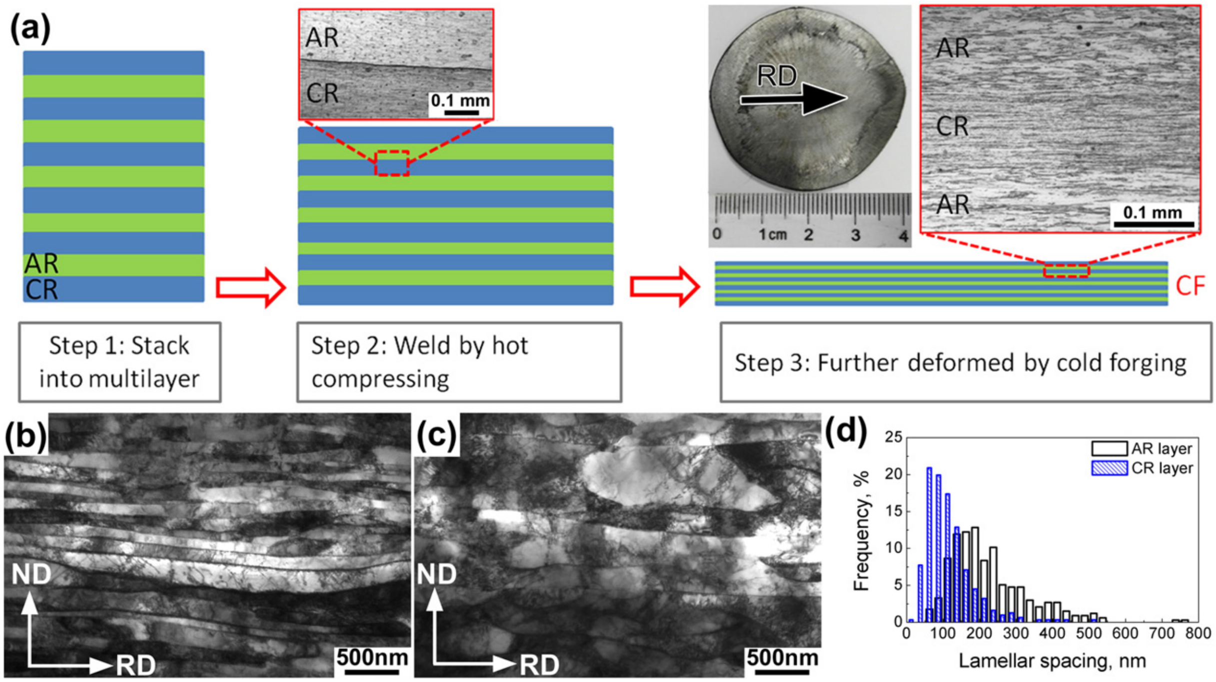


**Fig. 1.** (a) Schematic illustration of the fabrication process of the IF steel before annealing. Optical microscopy images of the interfaces after compression and after cold forging are also included in step 2 and step 3. The cold forged plate has a diameter around 40 mm as shown in step 3. The original rolling direction of the CR plate is indicated by RD. TEM images of the CR layer (b) and the AR layer (c) obtained on the longitudinal section of a CF specimen. (d) Distance between lamellar boundaries measured along the original ND of the CF specimens. Totally more than 600 boundaries were counted in both the CR and AR layers.

mechanical properties. Dog-bone shaped tensile samples with a gage length of 12 mm and a width of 2.5 mm were used. Electron back-scatter diffraction (EBSD) using an Oxford Aztec detector attached to a JEOL 7800F scanning electron microscope (SEM) was applied to characterize the microstructures of the annealed specimens. Detailed microstructural characterization of the deformed sample were done in a JEOL 2100 transmission electron microscope (TEM). All the microstructural observations were conducted on the original longitudinal section (RD-ND plane).

The microstructures of the IF steel after cold forging is shown in Fig. 1(b) and (c). It is seen that both the original CR and AR layers have lamellar dislocation boundaries, which are typical for IF steel deformed to high strains [10]. The average boundary spacings along the original ND are 116 nm and 226 nm in the CR layer and the AR layer, respectively.

As an example, the EBSD maps of the 600 °C 1 h and 575 °C 3 h annealed samples are shown in Fig. 2. Four layers are included in Fig. 2(a), in which only high angle boundaries (≥15°) are shown. On

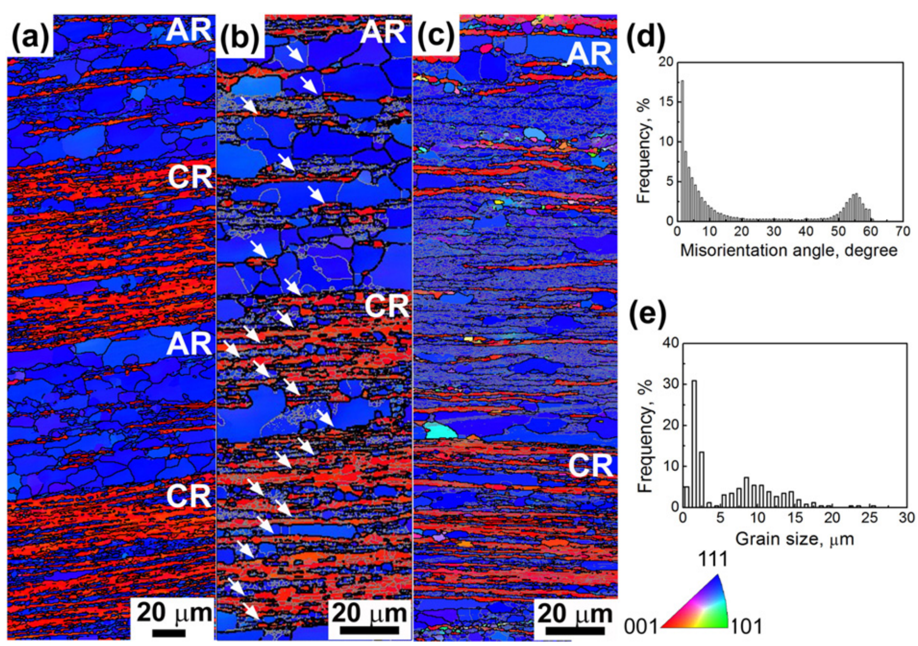


**Fig. 2.** Examples of EBSD mapping of the sample annealed at 600 °C for 1 h (a) and (b); and 575 °C for 3 h (c).Thick black lines represent high angle boundaries and thin white lines indicate low angle boundaries. (d) and (e) Corresponding misorientation angle distribution and grain size/boundary spacing distribution of the 600 °C annealed sample. In order to show the bimodal grain size distribution, same number of grains/boundaries was counted for coarse and fine grain size/boundary spacing.

first sight, “coarse grain” layer with dominate blue color (ND aligned along 〈111〉 direction, AR layer) and “elongated grain” layer mainly with red color (ND aligned along 〈001〉 direction, CR layer) can be recognized easily. This structure difference is inherited from the original stacking layers. In Fig. 2(b) and (c), only two layers are shown including details of low angle boundaries. A micro-scale alternation in grain size and orientations can be seen in these figures, as indicated by arrows in Fig. 2(b) for the lamellar grains in red color. Most of the coarse grains are surrounded by high angle boundaries (thick black lines) while a large amount of lamellae are surrounded by low angle boundaries (thin white lines). Similar microstructural feature can be found in Fig. 2(c) of the 575 °C annealed sample, except that a large amount of low angle grain boundaries is retained in the AR layer, while the CR layer has more high angle boundaries. Since the accumulative strain in the CR layer is higher than that in the AR layer, the CR layer should have a higher driving force for microstructural recovery and recrystallization during annealing. However, it is noticed that the thermal stability of the CR layer is more stable, differing from the homogeneous ultra-fine grained IF steel such as prepared by equal channel angular pressing [17]. By comparing Fig. 2(b) with Fig. 2(c), it is found that the grains in blue recover/recrystalize faster than that of the grains in red in both of the AR and CR layers. It is thus speculated that the texture and layer structure have effects on the thermal stability, which will be studied in our future work. Bimodal distributions of misorientation angle and grain size of the 600 °C annealed sample is shown in Fig. 2(d) and (e). The average grain sizes of small grains and coarse grains are 1.6 μm and 10.8 μm, respectively. This characteristic hierarchical layered structure plus heterogeneous lamella structure is thus called as L2 (layer + lamella) structure, which differs from the reported heterogeneous lamella structure [8], bimodal structure [2] and gradient structure [7].

Representative engineering stress-strain curves (S-S curve) of the annealed samples are shown in Fig. 3(a). The S-S curves of the CF, AR and CR samples are also plotted. The CF sample has almost no uniform elongation but the strength slightly increased, compared to the original CR sample, due to the higher deformation. Annealing after deformation usually initiates recovery and recrystallization, leading to an increase in ductility and a reduction in strength. A combination of high strength and large uniform elongation are achieved after annealing at 575 °C to 625 °C. A yield strength of 600 MPa and a uniform elongation about 10% is obtained after annealing at 575 °C for 3 h; and 500 MPa yield strength and 20% uniform elongation after annealing at 600 °C for 1 h. Even after annealing at 625 °C for 0.5 h, the uniform elongation is about 30% and the yield strength is about twice that of the AR sample.

Fig. 3(b) shows the work hardening rate - true strain curves. The work hardening rates of the CF, 575 °C and 600 °C annealed samples drop quickly after yielding, followed by a plateau in the regime of small strains and then decreasing continuously at higher strains. The CF and the 575 °C annealed samples show similar behavior of work hardening, which is thought to be related to the large faction of deformed structure. The evolution of work hardening rate of the 600 °C annealed sample is similar to that of the IF steel with a gradient structure and the Ti with a heterogeneous lamella structure [6,8,11]. It is speculated that the extra hardening behavior is caused by the mechanical incompatibility in the heterogeneous structure inducing multiaxial stresses and plastic strain gradient [12–14] during the elastic-plastic transition period. The work hardening rate smaller than 2500 MPa is zoomed in and shown in the inset of Fig. 3(b). The true stress-true strain curves are also plotted for checking the intersecting point for instability, according to the Consideré criterion [1] for necking. It is found that the CF sample quickly fractured after necking (intersected at a strain smaller than 0.05) while a large elongation (from a true strain of 0.10 to 0.2) can be achieved in the annealed samples by delaying premature localized necking and/or resisting cracking.

Fig. 4 shows the yield strength and uniform elongation combination obtained in the present study and other references [15–19]. It is clear that the combination of strength and elongation in the present study

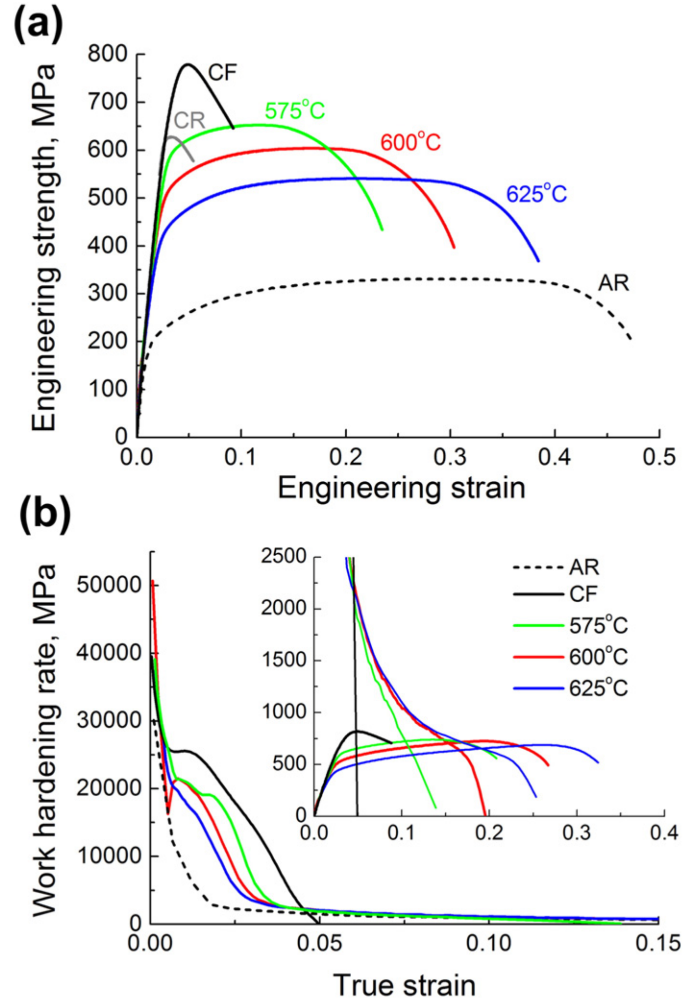


**Fig. 3.** (a) Typical engineering stress-strain curves of the cold forged and annealed samples. The tensile curves of the AR and CR samples are also plotted for comparison. (b) Work hardening rate-true strain curves. The inset figure depicts the intersection between the true stress-strain curves and the work hardening rate curves, which indicate the onset of necking according to the Consideré criterion.

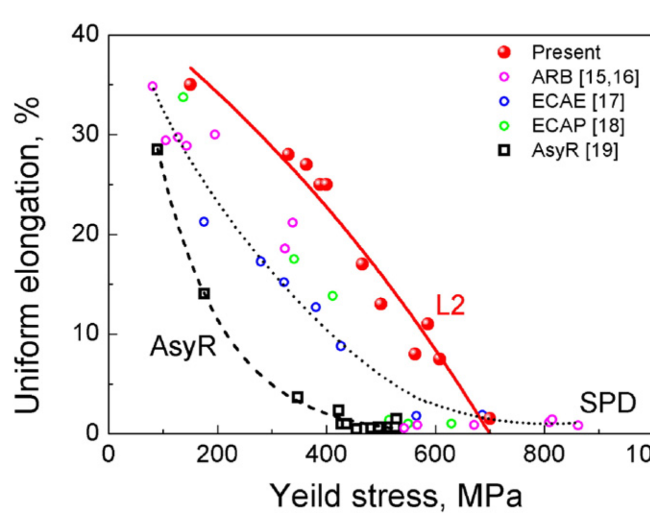


**Fig. 4.** Comparison of yield strength and uniform elongation combination between the present study and other data obtained from the references. The uniform elongation was determined from the engineering stress-strain curves in the present study.

is superior to other reported data, for example homogeneous ultra-fine grained IF steel prepared by accumulative roll bonding (ARB) [15,16] and equal-channel angular pressing/extrusion (ECAP/ECAE) [17,18] or heterogeneous structure by asymmetric rolling (AsyR) [19].

It is necessary to mention that, although various lamella structures have been investigated, seldom researches have tried the same material. For example, copper with bronze [20], Cu–10Zn with Cu [21], Nb with Cu [22–24], etc. were stacked to produce laminated structures. Cu/Cu laminated composite [9] was found to yield good toughness and fatigue properties but the effect of the microstructure was not discussed. In our research, we found that our new strategy can yield very good combination of strength and ductility by utilizing the heterogeneities in deformation strain of the same material. The superior mechanical properties of the L2 structured IF steel may be attributed to the following three factors:

(i) Hierarchical layer (~100 μm in this case), across which there are heterogeneities in grain size and texture.

Interfaces were suggested to be responsible for the observed high strain hardening and ductility in laminate and gradient materials [11,21,23]. Earlier reports on copper/bronze laminates have observed an interface-affected zone on microscale regardless of varying interface spacing [20]. Interfaces affect adjacent layers during deformation and promotes back stress hardening by producing more geometrically necessary dislocation pile-ups. Geometrically necessary dislocations accommodate the lattice curvature that arises whenever there is a non-uniform plastic deformation [13]. Geometrically necessary dislocations are generated to accommodate the complex strain and stress state induced by mechanical incompatibility, and promote the work hardening capability [11,21,23,25,26].

(ii) Heterogeneous lamella (~ 10 μm in this case), i.e. non-homogeneous distribution of grain within the layers.

Zeng et al. [14] have investigated the gradient nano-grained Cu by crystal plasticity finite element modeling. They found that spatial gradients arise due to progressive attainment of yield points in grains with gradient sizes and accordingly gradient slip resistances. Nix and Gao found that 5–12 μm as a length scale in Cu with which any strain gradient need to be considered [27]. Our micro-scale lamella structure, both in grain size, grain orientation and lamellar size as indicated by arrows in Fig. 2(d), is just within this level. Heterogeneous spatial distributions of gradient stresses and gradient plastic strains can lead to extra strain hardening in the material.

(iii) As a unique integral, it can be deduced that the ultra-fine grains impart high strength while the large grains stabilize the tensile elongation of the material. Combining the former mentioned spatial non-uniform strain and stress strengthening effect, high strength and good ductility can be achieved at the same time.

Furthermore, it's hypothesized that there exists an optimum interface spacing, comparable to the width of interface-affected zone and optimum volume fraction of fine/coarse grains with optimum size combination for the highest ductility without sacrificing the strength. Due to the inherited hierarchical structure character, the initial material, layer thickness and sample stacking sequence etc. can affect the heterogeneous structure and the mechanical properties may be adjusted by changing these parameters. Systematic studies on the mentioned parameters on microstructures and mechanical properties, underlining mechanisms and large-scale production are necessary in the future. It is believed that even better properties may be achieved by tailoring the L2 structure into better combination. Also, the plausibility of this strategy on other material systems is worth of investigation. Our study is just a testify for further developments.

The characteristic hierarchical layer + lamella structured, i.e. L2 structured, IF steel was produced by alternative stacking cold rolled and annealed IF steel followed by deformation and post-annealing. Both yield/ultimate strength and ductility in uniaxial tension of the L2 structured IF steel are greatly improved, which can be primarily attributed to the effect of interfaces and heterogeneous grain distribution due to mechanical incompatibility across layer interfaces and micro-scale lamellae. By tailoring the parameters affecting the L2 structure, even better properties may be achieved.

## Acknowledgments

This work was supported by the National Natural Science Foundation of China (51541101, 51671039, 51471039) and State Key Research and Development Program of MOST of China (2016YFB0700401, 2016YFB0700403). DJJ thanks the support of the "111" Project (B16007) by the Ministry of Education and the State Administration of Foreign Experts Affairs of China.

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