Achieving an excellent combination of strength and plasticity in a low carbon steel through dynamic plastic deformation and subsequent annealing

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**Abstract**

An investigation was conducted to evaluate the effect of dynamic plastic deformation (DPD) and post-DPD annealing on the microstructural evolution and mechanical properties of a tempered low carbon-low alloy steel. The results showed that ultrafine-grained structures consisting of elongated martensitic laths and sub-grains are achieved after DPD processing. The amounts and sizes of carbides in the steels, identified as (Fe, Cr, Mn, Mo)3C, decreased markedly with DPD straining due to their fragmentation and dissolution but the strength increased and the plasticity showed a slight decrease with increasing DPD strain. A simultaneous improvement in the strength and plasticity was obtained at strains below ~0.8. This increase in strength by ~30-60% after DPD processing by comparison with the as-received state is attributed primarily to grain boundary strengthening and dislocation strengthening and the good plasticity is due to the occurrence of more active sliding systems and a reduction in the stress concentration during loading because of a decrease in the amounts of M3C distributed along the interfaces. After post-DPD annealing both the strength and the plasticity improved by comparison with the as-received steel in the tempered state. Strengths higher by ~15-35% than the as-received condition were attributed to a combination of grain boundary strengthening, dislocation strengthening and precipitation strengthening derived from a re-precipitation of fine and dispersed carbides. The dislocation recovery occurring during annealing led to a decrease in strength compared with the strength before annealing. The incremental increase in plasticity after annealing is ascribed to dislocation recovery and the inhibition in micro-crack propagation due to an even distribution of fine carbides within the matrix.

**Key words:** Annealing; Dynamic plastic deformation; Low carbon steel; Plasticity; Strengthening mechanism.

1. **Introduction**

A refining of the microstructure to obtain an ultrafine-grained (UFG) structure is an effective procedure for strengthening metallic materials [1-4]. Furthermore, the use of dynamic plastic deformation (DPD) is a significant processing method for producing UFG structures through severe deformation at rapid strain rates of the order of 102 - 103 s-1 [2, 5-8]. By comparison with conventional severe plastic deformation (SPD) techniques, such as equal channel angular pressing (ECAP) [8a,9-11], high pressure torsion (HPT) [11a,12, 13] and accumulative roll bonding (ARB) [14, 15], DPD was shown earlier to be more effective in refining the microstructure by introducing exceptionally finely-spaced boundaries [8, 16]. Several DPD processing methods are now available including the use of the traditional split Hopkinson pressure bar (SHPB) [8, 17], high-speed hammer impacting [18], dynamic channel angular pressing (DCAP) [19] and dynamic high-pressure torsion (DHPT) [20]. Of these various DPD procedures, the use of SHPB is especially attractive because it has a demonstrated much higher efficiency in producing UFG structures at high strain rates of the order of ~103 s-1 [21].

In practice, the strength-plasticity paradox is a major challenge in the research and development of new materials [22-24]. Most studies have shown that the strength enhancement of DPD-processed steels is accompanied by a drastic reduction in plasticity [25-27][25]. For example, the strength of a 9Cr1Mo steel processed by DPD increased significantly from ~675 MPa for the as-received material to ~1247 MPa but the elongation decreased from ~21.5% to ~5.0% [26]. DPD processing also sharply reduced the elongation of a quenched low carbon steel from ~17.5% to ~3.8% [27]. In order to improve the plasticity of the processed steel, subsequent annealing may be conducted to eliminate the internal stresses produced by the deformation. For example, post-DPD annealing led to an improvement in plasticity of the 9Cr1Mo steel with the elongation increasing to ~10.9% while the tensile strength dropped by ~35% to ~436 MPa [26]. Nevertheless, the elongation remains reduced by comparison with the unprocessed value. A synchronous improvement in both strength and plasticity of a 9Cr1Mo steel with a strong carbide-forming element (W, V, Ta) was obtained using SPD in cold forging to a reduction of 94% and subsequent annealing at 700℃ [28]. The improved elongation of ~18%-23% was attributed to the re-precipitation of finer and uniformly distributed carbides during the annealing process. However, it should be noted that the strength increased by less than ~20% to ~120 MPa at such a large strain due to a complete recrystallization during the high temperature annealing.

Tempered low carbon-low alloy steels are widely used in automobile, bridge and other engineering fields due to their combination of high strength, good plasticity and toughness, excellent weldability and generally low cost [29-32]. With the rapid development of numerous engineering applications, it is becoming increasingly important to achieve improved mechanical properties in these steels. In practice, earlier studies demonstrated that DPD processing may improve the strength of the steel more significantly compared with using SPD procedures at low strain rates [33] and additional low temperature annealing below the recrystallization temperature contributes to retaining the high strength of the DPD-processed steels because of the absence of complete recrystallization [26]. Simultaneously, the re-precipitation of fine carbides during annealing is conducive in improving the deformability of the steel [28, 34]. Thus, it is reasonable to anticipate that an excellent combination of strength and plasticity may be achieved in a low carbon steel with micro-alloying of carbide-forming elements such as Cr, Mo and V by using DPD processing and appropriate subsequent annealing. Nevertheless, there are no reports to date demonstrating the validity of this process.

Accordingly, the present investigation was initiated specifically to evaluate the influence of DPD processing and post-DPD annealing at the low temperature of 450℃ on the microstructural evolution and mechanical properties of a cost-saving low carbon-low alloy steel.

**2. Experimental material and procedures**

*2.1. Material and preparation*

A low carbon steel with a chemical composition of Fe-0.13C-0.23Si-1.09Mn-0.6(Cr+Ni+Mo+V) in wt.% was received as a plate after a quenching treatment at 920℃ for 30 min, water cooling and then tempering at 550℃ for 1 h to obtain a tempered martensitic microstructure. The as-received material was cut into cylinders with diameters of 10 mm and lengths of 12 mm and then subjected to DPD at a strain rate of ~103 s-1 under uniaxial compression at room temperature (RT, ~25℃) using an SHPB facility described in detail in an earlier report [35] with input and output bars having diameters of 37 mm. The steels were pressed for 2, 4 and 6 times to reach strains of ~0.4, ~0.8 and ~1.2 where the strain was estimated as *ln(L0/Lf)*, where *L0* and *Lf* are the cylinder heights before and after DPD processing, respectively. The deformed specimens were then further annealed at 450℃ for 1 h to explore the annealing behavior of the DPD processed samples. For convenience, the steels processed by DPD for strains of 0.4, 0.8 and 1.2 are denoted DPD-0.4, DPD-0.8 and DPD-1.2 and the corresponding post-DPD annealed steels are designated DPD-0.4-450, DPD-0.8-450 and DPD-1.2-450, respectively. The DPD device and the samples before and after DPD processing are schematically illustrated in Figs.1 (a) and (b).

*2.2. Microstructural characterization*

The microstructures of the as-received, DPD-processed and post-DPD annealed samples were characterized using a scanning electron microscope (SEM, Hitachi-S4800), electron backscatter diffraction (EBSD, FEI Quanta 650F) and a transmission electron microscope (TEM, Tecnai F20). The microstructures were observed from the areas near the central part of the cross-section of each cylinder which was perpendicular to the pressing direction. The elemental distributions and compositions of precipitations were investigated using high-angle annular dark field STEM (HAADF-STEM) imaging with energy dispersive X-ray spectroscopy (EDS) equipped in TEM and 3D atom probe tomography (3DAP, LEAP 4000HR) [36, 37].

The samples for the SEM observations were mechanically polished and etched in a 3% nital solution. The EBSD analysis was carried out with a step size of 0.2 μm and the results were analyzed with Channel 5 software. Low-angle grain boundaries (LAGBs) were defined as having misorientation differences from 2° to 15° and high-angle grain boundaries (HAGBs) had misorientations larger than 15°. Additionally, maps of the Schmid factors were constructed based on crystalline orientations, thereby including both the “soft” grains with high Schmid factors which are favorably oriented for slip and the “hard” grains with low Schmid factors which are less favored for slip [38]. For TEM characterization, thin foils were prepared by twin-jet electro polishing in an electrolyte of 90% methanol and 10% perchloric acid with a voltage of 20V at 30℃. The widths of the martensitic laths and the sizes of sub-grains and precipitates were measured using Nano-measure software. The density of precipitates in the matrix was estimated using the relationship [39]:

(1)

where *f* is the volume fraction of precipitates in the microstructure, *N* is the number of precipitates in an observed area, *S* is the size of the observed area and *d* is the average size of the precipitates in terms of the diameter or side length.

*2.3. Mechanical properties testing*

The Rockwell C hardness (HRC) was taken from the cross-sections of the as-received, DPD processed and post-DPD annealed samples with the average HRC values calculated from 6 to 8 separate measurements. Tensile testing was carried out at RT (~298 K) at an initial strain rate of 5.0 × 10-4 s-1 using an Instron 50 KN tensile tester with a laser extensometer. Planar dog-bone tensile samples with gauge lengths of 5 mm were machined from the central parts of the samples. At least three samples were tested for each condition to confirm the reproducibility. Fig.1 (c) is a schematic diagram of the sample positions cut from the DPD-processed discs/cylinders for microstructural observations and tensile testing.

***3. Results***

*3.1 Initial microstructure before DPD processing*

Fig. 2 shows the microstructure of the initial as-received steel. Figs 2 (a)-(c) display carbides with different sizes distributed in the interfaces and the matrix of tempered martensitic laths having average widths of ~340 nm. Both the smaller sizes of ~40 nm dispersed in the matrix and the larger sizes over ~120 nm distributed along the lath boundaries were identified as M3C through Selected Area Electron Diffraction (SAED) patterns, High Resolution TEM (HRTEM) and subsequently Fast Fourier Transform (FFT)as in Fig. 2 (d). The EDS results in Fig. 2 (e) reveal that the M3C particles are rich in C, Cr, Mn and Mo. Fig.3 is a 3DAP analysis of the precipitates. Inspection of the isosurface diagrams (Fig. 3 a) shows that there are C, Cr, Mn and Mo elements segregated in cementite where this is consistent with the EDS results. The composition of the M3C was further proven to be (Fe,Cr,Mn,Mo)3C with an accurate at.% of 59Fe-11Cr-5.1Mn-0.9Mo-24C based on the line scanning results in Fig. 3 (b). In addition, Fig.2 (a) shows that after quenching and tempering the austenite grains are transformed and divided into several tempered martensitic substructures comprising parallel laths with an average size of ~20 μm.

*3.2* *Microstructures after DPD processing*

Microstructures of the DPD-processed steels are displayed in Fig. 4. The lath structures shown in Figs. 4 (a)-(c) are elongated and refined compared with the as-received material shown in Fig. 2 (a) and the martensitic substructures is hardly distinguishable at a strain higher than ~0.8. Further observation shows that two different sub-microstructures are formed during DPD processing and these are marked as zones 1 and 2 consisting of sub-grains/cells (Figs. 4 a1-c1) and elongated martensitic laths (Figs. 4 a2-c2), respectively. These two substructures are caused by a deformability difference between the laths having different orientations. A statistical analysis shows that the sub-grain/cell sizes decrease from ~550 to ~300 nm while the mean widths of the martensitic laths decrease from ~237 to ~120 nm with increasing DPD strains from 0.4 to 1.2.

The dark field TEM images in Figs.4 (d)-(f) in the right column show that the amounts and sizes of the precipitates in the matrices of the DPD-processed steels are significantly reduced compared with the as-received material shown in Fig. 2 (c). Table.1 summarizes the sizes and number densities of carbides in the as-received and DPD-processed steels, respectively. It is noted that the amounts and sizes of the M3C particles distributed both in the matrix and along the interfaces decrease with DPD straining.

The EBSD orientation color maps and corresponding IPF of the as-received and DPD-processed steels under various cumulative strains are shown in Fig. 5. Thus, Fig. 5 (a) shows that the grains are positioned in random crystallographic directions within the as-received steel and they exhibit a very weak texture with a peak intensity of ~1.65 MUD where MUD corresponds to multiples of uniform density. The size of the martensitic substructure is ~23.8 μm which is consistent with the SEM images in Fig. 2 (a). Inspection of Fig. 5 (b) shows that the martensitic substructures in the DPD-0.4 sample are clearly deformed and the average size is refined to ~16.2 μm. In addition, a preferred orientation of grains becomes apparent with a strong < 111 > fiber texture and a weak < 100 > fiber texture with a total intensity of ~4.02 MUD. The microstructures become further ultra-refined and elongated with indistinct grain boundaries as the DPD strain increases to 0.8 and 1.2 as shown in Figs. 5 (c) and (d) so that it becomes difficult to estimate an accurate substructure size using the EBSD method. Furthermore, the fibrous textures are further strengthened and the peak intensities of MUD reach ~5.09 and ~5.34 after DPD processing to strains of 0.8 and 1.2, respectively.

Fig. 6 displays the misorientation angles for the as-received and DPD-processed steels. It is readily apparent from Fig. 6 (a) that the as-received steel exhibits a bimodal distribution of misorientation angles with an LAGB fraction of ~50.3%. With increasing DPD strains from 0.4 to 0.8, the bimodal distribution of the misorientation angles is weakened and the fraction of LAGBs increases from ~63.6% to ~70.4% due to the formation of more sub-grains [8, 40], as depicted in Figs 6 (b) and (c). Fig. 6 (d) shows that a further increase in the DPD strain to 1.2 leads to a decrease in the fraction of LAGBs to ~65.8% which may be caused by the partial dynamic recrystallization occurring at higher strains [41, 42].

*3.3* *Microstructures after post-DPD annealing*

Fig. 7 depicts the SEM, bright and dark field TEM images of the steels after DPD and subsequent annealing at 450℃ showing there remain two different microstructures which are similar to the situation after DPD processing in Fig. (4). Figs.7 (a1)-(c1) present the equiaxed sub-grains/cells with distinct boundaries that transform from the sub-grains in Figs 4 (a1)-(c1) having sizes of ~557，~421 and ~296 nm for the DPD-0.4-450, DPD-0.8-450 and DPD-1.2-450 samples, respectively. The widths of the martensitic laths for the samples of DPD-0.4-450, DPD-0.8-450 and DPD-1.2-450 as shown in Figs 7 (a2)-(c2) are ~234, ~178 and ~127 nm, respectively. Both the sub-grain sizes and the martensitic lath widths are close to the values before annealing and therefore it is inferred that the post-DPD annealing at 450℃ has little effect on the sub-grain sizes or widths of the martensitic laths.

Further inspection of Figs 7 (a1)-(c1) and (a2)-(c2) shows that the dislocation density in the DPD-processed steel after annealing is significantly reduced by comparison with that before annealing as shown in Figs 4 (a1)-(c1) and (a2)-(c2) and some M3C particles, as marked by arrows, are distributed along the martensitic lath boundaries and sub-grain boundaries. In addition, large numbers of fine M3C carbides are distributed uniformly in the matrix shown in Figs 7 (d)-(f). The sizes and densities of the carbides in the post-DPD annealed steels are summarized in Table 2. This summary shows that increasing the DPD strain from 0.4 to 1.2 leads to an incremental increase in the density and a decrease in the size of the M3Calong the interfaces of the annealed steels, while both the density and the size of the M3Cdistributed within the matrix decline with DPD straining. In addition, it is readily apparent that the number densities of the precipitates with finer sizes increase markedly compared with that before annealing as listed in Table.1.

Fig. 8 presents typical EBSD orientation color maps with the corresponding IPF and histograms of the misorientation angle distributions for the post-DPD annealed steels. As depicted in Figs 8 (a), (c) and (e), a strong <111> fiber texture and a weak < 100 > fiber texture are observed in the three annealed samples and this is similar to the steels before annealing as shown in Figs 5 (b)-(d). The peak MUDs of the texture for the post-DPD-(0.4/0.8/1.2) annealed steels are ~3.87, ~5.00 and ~4.98, respectively, and the LAGB fractions in the annealed steels are ~60.5%, ~68.3% and ~64.8%, respectively, as displayed in Figs 8 (b), (d) and (f). It is readily apparent that both the texture intensities and the LAGB fractions decrease slightly after annealing compared with the situation before annealing.

*3.4 Evaluation of the Schmid factors before and after annealing*

Fig. 9 shows the Schmid factor distribution maps with average Schmid factor values for the as-received, DPD-0.4 and DPD-0.4-450 samples. A large area of light-colored zones with low Schmid factors is present in the as-received samples and the average Schmid factor of the grains is ~0.42 as displayed in Fig. 9 (a). After DPD processing at a strain of 0.4, the light-colored area is reduced and the Schmid factor increases to ~0.45 as shown in Fig. 9 (b). Fig. 9 (c) indicates that post-DPD annealing reduces the size of the light-colored region and further increases the Schmid factor to ~0.47. Thus, the results show that the Schmid factors are increased by DPD processing and further post-DPD annealing.

*3.5 Mechanical properties of DPD-processed and annealing-treated samples*

Fig. 10 shows the true stress-true strain curves of the as-received, DPD-processed and post-DPD annealed steels. Fig. 11 intuitively presents the effect of the DPD strain on the mechanical properties as extracted from Fig. 10. In Figs 11 (a) and (b) it is apparent that the mechanical properties, including hardness (HRC) and yield strength (*σ0.2*), of both the DPD-processed and post-DPD annealed steels are effectively improved by comparison with those of the as-received steel and, in addition, the HRC together with the σ0.2 are significantlyenhanced with DPD straining. Specifically, the *σ0.2* increases by ~30-60% (~248-471MPa) with the DPD strain increasing from 0.4 to 1.2 by comparison with the tempered steel. A post-DPD annealing gives a reduction in the tensile strength and hardness compared with that before annealing. For example, the yield strengths of the post-DPD annealed steels are lower by ~140-180 MPa than their DPD-processed counterparts but they are superior by ~15-35% (~115-288 MPa) to the as-received condition.

The effects of DPD processing and post-DPD annealing on the overall elongations of the steels are shown in Fig. 11 (c) where the elongations of the steels after processing by DPD at strain up to 0.8 are higher than for the as-received steel with ~15.5% but slightly reduced to ~13.3% for the DPD-1.2 steel. After post-DPD annealing, the plasticity is improved compared with the DPD-processed samples. Thus, the elongations of the DPD-0.4-450, DPD-0.8-450 and DPD-1.2-450 samples are higher by ~0.6%, ~1.3% and ~3.1%, respectively, compared with their counterparts before annealing. It is also noticeable that the plasticity of the post-DPD annealed steels at all experimental strains are better than for the as-received steel.

In addition, the true stress-true strain curves in Fig.10 (a) show that the flow stresses decrease immediately with increasing strain after reaching a maximum point in the true strain of no more than 0.01 in the DPD-processed steels, thereby exhibiting a negative strain hardening ability. By contrast, a strain hardening stage and uniform elongations appear again in the true stress-strain curves of the post-DPD annealed specimens in Fig. 10 (b) where this is similar to the as-received steel. Consequently, an excellent combination of strength, plasticity and strain hardening ability of the steels may be achieved through DPD and a post-DPD annealing treatment under the present experimental conditions.

Fig.12 exhibits the microstructures of the longitudinal sections near the fracture surfaces in the as-received and post-DPD annealed steels after tensile testing. Many micro-cracks propagate along the martensitic laths and the interfaces between the matrix and the carbides in the as-received steel as observed in Fig. 12 (a) due to the localized stress concentration arising at these zones from dislocation accumulation. By contrast, there are only a few micro-cracks in the post-DPD annealed samples as shown in Fig.12 (b) and this appears to be due to the finer carbides in the post-DPD annealed steels that effectively reduce the degree of concentrated stresses. In addition, inspection of the higher magnification image in Fig. 12 (c) reveals that the fine and evenly dispersed carbides can also effectively retard the initiation and propagation of micro-cracks under tensile loading.

***4. Discussion***

*4.1 The mechanism of grain and carbide refinement during DPD processing*

Fig.4 shows that the microstructure of the steel after DPD processing consists of two different morphologies: elongated tempered martensitic laths and fine sub-grains/cells formed in the refined laths. This is due to the inhomogeneous deformation of the steel during DPD processing which is a consequence of the different orientations of the martensitic laths. It is relatively difficult for the laths with hard orientations to deform so that they are only elongated during DPD processing due to the occurrence of less deformation whereas the laths with soft orientations having slip planes parallel or roughly parallel to the deformation direction are prone to severe deformation and gradually form fine sub-grains/cells under the action of dislocations [43, 44]. As shown in Fig.4 (a1), dislocation tangles (DTs) are formed in the original martensitic laths through this dislocation movement together with an interaction between the accumulated dislocations and the carbides in the initial stage of DPD processing and thereafter dense dislocation walls (DDWs) form with increasing DPD strain. Finally, these DDWs evolve into fine cellular substructures as the strain further increases (Fig.4 (c1)). In addition, the slip systems of these fine grains turn to the main deformation direction with straining, leading to increasing textures in the DPD-processed steels as demonstrated in Fig.4.

As listed in Table.1, the sizes and number densities of the carbides are reduced with increasing DPD strain and this is attributed to the fragmentation and dissolution of the carbides during deformation. During DPD processing, fragmentation occurs due to dislocation tangling and the piling of dislocations at the carbides (Fig.4 (a2)) since the carbides are usually defined as the brittle phase due to their limited slip systems [45]. Then the fine carbides dissolve to the matrix spontaneously because the carbon atoms enriched by high densities of dislocations have lower internal energies for the low-carbon steel than the very small carbides pre-existing in the matrix. In addition, the dissolution of the broken carbides is further accelerated because the high density dislocations introduced by DPD processing may act as fast channel for carbon atom diffusion from dissolved precipitates into the matrix [46, 47].

*4.2 Microstructure evolution during post-DPD annealing*

After post-DPD annealing, the sub-grains in steels became equiaxed with clearer and better defined boundaries than before annealing (Figs.7 a1-c1). This indicates that recrystallization occurs at 450℃ because of the high stored energy in the DPD-processed samples even though the annealing temperature is a little lower than the conventional recrystallization temperature of ~0.3-0.4Tm, where Tm is the absolute melting temperature for the steel [48]. This recrystallization process also reduces the fractions of LAGBs in the annealed steels by comparison with the DPD-processed steels as shown in Fig. 8. Moreover, the sub-grain sizes and the martensitic lath widths remain almost unchanged compared with the sizes before annealing due to the pinning effect of fine precipitates on the grain boundaries.

Fig. 7 and Table 2 demonstrate that numerous finer carbides re-precipitate in the post-DPD annealed steels. The dissolution of carbides during DPD processing leads to the supersaturation of carbon atoms and other carbide-forming elements within the matrix while the high density of dislocations and sub-grain boundaries provide sufficient numbers of nucleation sites for the re-precipitation of carbides during post-DPD annealing [49]. Thus, more and finer carbides are visible in Fig.7 (d-f) by comparison with Fig.4 (d-f). Additionally, more M3C are formed along the grain boundaries than in the matrix with increasing DPD strain from 0.4 to 1.2 after post-DPD annealing, and this is due to the presence of more interfaces in the microstructure after DPD at higher strains which promotes the precipitation of M3C during annealing.

*4.3 Mechanisms for the enhanced hardness and tensile strength*

The hardness and tensile strength of the DPD-processed and post-DPD annealed steels are significantly enhanced compared with the as-received steel as shown in Fig. 11. This is due to a combination of several strengthening mechanisms. Firstly, there is a strong grain boundary strengthening effect in the DPD-processed and post-DPD annealed steels. Sub-grains and finer martensitic laths are formed after DPD and retained during post-DPD annealing compared with the as-received steel, and the sub-grain size and martensitic lath width further decrease with increasing DPD strain as shown in Fig. 4 and Fig. 7. According to the Hall-Petch relationship, the refined microstructures make contributions to the enhancement of hardness and strength [50,50a]. Then a comparison of the TEM images in Figs 2 and 4 confirms that the dislocation densities in the DPD-processed samples are much higher than in the as-received steel, thereby producing a stronger dislocation strengthening effect. Dislocation recovery during post-DPD annealing leads to a strength reduction in the annealed samples by comparison with the DPD-processed steels (Fig. 11). However, a reasonable level of dislocation strengthening is retained in the annealed steels because the dislocation recovery is delayed by the pinning effect of the re-precipitated fine carbides, as demonstrated in Fig. 7.

Finally, a precipitation strengthening mechanism should be considered if carbides are present where this strengthening effect is closely relevant to the size and volume fractions of the precipitates based on the Orowan looping mechanism [51, 52]. It is reasonable to infer, therefore, that the precipitation strengthening in DPD-processed steels is weakened compared with the as-received steel as the precipitate densities decrease drastically because of the precipitate dissolution during DPD processing. However, precipitation strengthening cannot be neglected even after post-DPD annealing since large numbers of finer carbides are distributed homogeneously in the matrix due to their re-precipitation.

In conclusion, therefore, the improved strength of the DPD-processed steels is mainly attributed to grain boundary strengthening and dislocation strengthening while a combination of grain boundary strengthening, precipitation strengthening and dislocation strengthening contribute also to an enhancement in strength for the post-DPD annealed steels.

*4.4 Mechanisms for the improved plasticity after DPD and post-DPD annealing*

It is especially impressive that the steels after DPD processing maintain good plasticity when the strength is enhanced since this is contrary to earlier studies where strength improvements generally led to reductions in plasticity [22-27, 33, 34]. As shown in Fig. 11 (c), elongations of the DPD-processed steels are even higher than for the as-received samples at strains lower than ~0.8. In practice, the plasticity depends mainly on parameters such as the active sliding systems [53, 54]. Fig. 9 shows that DPD processing at a strain of 0.4 increases the Schmid factor to ~0.45 from ~0.42 in the as-received steel. Furthermore, the frequency distribution histogram of the Schmid factors shown in Fig.13 reveals that the fraction of grains with Schmid factors between ~0.47-0.50 becomes larger (~51.1%) in the DPD-0.4 steel compared with the as-received material (~39.4%). Hence, the steels after DPD for strain of 0.4 have even better deformability [53]. In addition, plasticity improvements in the steels after DPD processing may also be related to a reduction in the presence of carbides and especially in a decrease in the M3C distributed along the interfaces between the martensitic laths which effectively reduces the possibility of a stress concentration associated with these particles during tensile deformations [55]. Some other factors, including the appearance of <111> fiber texture [56, 57] and the increasing fraction of LAGBs, are also conducive to an improvement in plasticity [58, 59] but the higher dislocation density in the steel after DPD for a strain of ~1.2 leads to an elongation which is slightly lower than for the as-received and DPD-0.8 steel.

Post-DPD annealing produces a superior deformability of the steels compared to the as-received and DPD-processed samples as shown in Fig. 11 (c). Thus, in Fig. 9 (c) and Fig. 13, the average Schmid factor increases to ~0.47 and the fractions of grains having Schmid factors higher than 0.47 reached to ~58.2% after annealing in the DPD-0.4 steel, thereby indicating an improved plasticity for the annealed steels. In addition, the dislocation density decreases gradually as a result of the occurrence of recovery during post-DPD annealing, and this makes it easier for mobile dislocation movement during tensile testing. The numerous finer re-precipitated carbides in the post-DPD annealed steels strengthen the matrix and effectively inhibit the initiation and propagation of micro-cracks under tensile loading (Fig. 12 (c)) so that a better strain hardening ability and deformability are achieved [28]. To summarize, the further improved plasticity of the post-DPD annealed steels is derived from a combination of more active sliding systems, a decreasing dislocation density and a dispersed distribution of finer carbides.

**5. Summary and conclusions**

1. Experiments were conducted to investigate the microstructural evolution and mechanical properties of a tempered low carbon-low alloy steel after DPD processing for strains in the range of ~0.4-1.2 and post-DPD annealing at 450℃ .

2. DPD processing produces ultrafine-grained structures consisting of elongated martensitic laths with average widths of ~120 nm and sub-grains formed with mean sizes of ~300 nm after DPD for a strain of 1.2. With increasing DPD strain, the amounts and sizes of the carbides in the matrix, identified as (Fe, Cr, Mn, Mo)3C, decrease markedly due to a fragmentation and dissolution during DPD processing. Post-DPD annealing has no effect on the sizes of the martensitic laths and the sub-grains but it leads to a re-precipitation of a large number of finer and dispersed carbides because of the nucleation sites provided by the high densities of dislocations and sub-grain boundaries introduced by the DPD processing.

3. DPD processing for strains up to ~0.8 improves the strength and plasticity simultaneously over the as-received tempered condition. With increasing strain, the strength increases and the plasticity slightly decreases. The increased strength by ~30-60% after DPD processing is due mainly to grain boundary strengthening and dislocation strengthening, whereas the good plasticity is due to the presence of more active sliding systems and the reduction of stress concentration during loading because of a decrease in the M3C distributed along the interfaces between the martensitic laths.

4. Post-DPD annealing leads to a decrease in strength and an increase in plasticity compared with before annealing. However, both the strength and plasticity improve after post-DPD annealing by comparison with the as-received steel. The enhancement of strength by 15~35% is due to a combined effect of grain boundary strengthening, precipitation strengthening and dislocation strengthening. The increment in plasticity after annealing is attributed to the reduction in the dislocation density caused by recovery and an inhibition in micro-crack propagation due to the even distribution of fine carbides within the matrix.

4. The results demonstrate that an excellent combination of strength, plasticity and strain hardening may be achieved in the experimental steel through DPD and post-DPD annealing.

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**Figure Captions：**

Fig.1. Schematic diagram of (a) DPD device, (b) samples before and after DPD processing and (c) position for microstructure observation and tensile testing

Fig.2. Microstructures of the as-received steel:(a) SEM image, (b)-(c) bright field and corresponding dark field TEM images with SAED pattern of M3C along interface, (d) HRTEM image and FFT analysis of M3C in matrix, (e) HAADF-STEM image with EDS results of M3C particles

Fig.3. 3DAP analysis of the precipitates in the as-received steel, (a) isosurface of C, Mn, Cr and Mo elements, (b) APT composition measurement of a carbide particle

Fig.4. Microstructures of the steel after DPD processing at strain of (a, a1, a2, d) 0.4, (b, b1, b2, e) 0.8 and (c, c1, c2, f) 1.2. Herein (a, b, c) showing SEM images, (a1, b1, c1) and (a2, b2, c2) showing bright field TEM images corresponding to the zones marked 1 and 2 in (a, b, c), respectively, and (d, e, f) showing precipitates in dark field TEM images.

Fig.5. EBSD orientation color maps and corresponding IPF of the steel in (a) the as-received condition and after DPD processing at strain of (b) 0.4, (c) 0.8 and (d) 1.2

Fig.6. Histogram of the misorientation angle distributions for the steels in (a) the as-received condition and after DPD processing at strain of (b) 0.4, (c) 0.8 and (d) 1.2

Fig.7. Microstructures of the steel after post-DPD annealing at strain of (a, a1, a2, d) 0.4, (b, b1, b2, e) 0.8 and (c, c1, c2, f) 1.2, where (a, b, c) show SEM images, (a1, b1, c1) and (a2, b2, c2) showing bright field TEM images corresponding to the zones marked 1 and 2 in (a, b, c), respectively, and (d, e, f) showing precipitates in dark field TEM images

Fig.8. EBSD orientation color maps with corresponding IPF and histogram of the misorientation angle distributions for (a)-(b) DPD-0.4-450, (c)-(d) DPD-0.8-450 and (e)-(f) DPD-1.2-450 steels

Fig.9. Schmid factor distribution maps of (a) the as-received, (b) DPD-0.4, (c) DPD-0.4-450 samples

Fig.10. Mechanical properties of (a) as-received and DPD processed and (b) post-DPD annealed samples

Fig.11. The influence of DPD strain on the (a) yield strengths, (b) HRC and (c) elongations of the DPD processed and post-DPD annealed steels

Fig.12. Longitudinal sections near the fracture surfaces of (a) the as-received and (b), (c) the post-annealed samples after tensile testing

Fig.13. Frequency distribution histogram of Schmid factors for the as-received, DPD-0.4 and DPD-0.4-450 steels

**Table Legends：**

Table.1 Sizes and densities of precipitates in DPD-processed steels

Table.2. Sizes and densities of precipitates in post-DPD annealed steels