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Unconfined Twist: a Simple Method to Prepare Ultrafine Grained Metallic Materials

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ABSTRACT

A new simple method – unconfined twist was employed to prepare ultrafine grained (UFG) Fe wire. A coarse grained (CG) Fe wire with a diameter of 0.85 mm was fixed at one end, and twisted at the other end. After maximum twist before fracture, in the cross-sectional plane, concentrically deformed layers with a width of several micrometers formed surrounding the center axis of the wire. The near-surface deformed layers consist of lamella grains with a width in submicrometer range. In the longitudinal plane, deformed bands (with a width of several micrometers) formed uniformly, which were composed of lamella crystallites (with a width in submicrometer range). The tensile yield strength and ultimate strength of the twisted Fe wire are increased by about 150 % and 100 % compared with the values of its CG counterpart.

INTRODUCTION

Ultra-fine grained (UFG) metals and alloys have been the subject of considerable research in recent years. Such interest has been spurred by the recognition that these materials possess some appealing mechanical properties, such as high yield and fracture strengths, superior wear resistance, increased resistance to tribological and environmentally-resisted damage, increased strength and/or ductility with increasing strain rate and potential for enhanced superplastic deformation at lower temperature and higher strain rates. Nevertheless, the industrial application of the UFG materials has not been realized yet because of the difficulty to fabricate them into dense and bulk dimensions. Among the numerous UFG material synthesis methods, such as mechanical alloying and compaction [1,2], inert-gas condensation and consolidation [3], electrodeposition [4], crystallization of amorphous solids [5,6] and severe plastic deformation (SPD) [7], the SPD method, such as equal channel angular pressing (ECAP) [8,9] is especially attractive because it can economically produce relatively large bulk dense UFG samples without changing their cross-sectional dimensions. Another SPD method, high-pressure torsion (HPT) [7], can effectively refine coarse grains into several tens nanometers, but it can only process very thin disk (about 1 mm thick). Moreover, both HPT and ECAP methods need very high pressure (up to several GPa). Such high pressure brought difficulties for the industrial applications of these SPD preparations. Therefore, simpler SPD method would be attractive.

In the present work, we developed a new simple SPD method – unconfined twist to process UFG metallic materials. In this method, an initial rod-shaped sample was fixed at one end, and was twisted at the other end. During the twist, severe plastic deformation occurred within the sample and formed the UFG structures. The twisted UFG sample is found to have greatly enhanced mechanical strength compared with its coarse grained (CG) counterpart.

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EXPERIMENTAL DETAILS

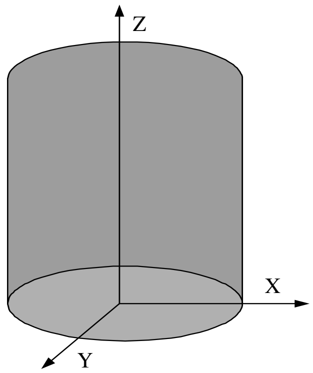

Figure 1. Schematical representation of the Fe wire.

Commercial Fe wire (with a diameter of 0.85 mm and a length of 12 mm) was fixed on a twisting machine and twisted for 180 rotations clockwise at a speed of 30 rpm. Tensile tests of the twisted and untwisted samples were carried out using a Shimadzu Universal Tester at a displacement rate of 1×10^{-2} mm/s. The morphology of the fracture surface was observed by JEOL Scanning Microscope 6300 FXV operating at an accelerate voltage of 30 kV.

The twisted UFG and initial CG Fe wires were then cut and polished cross-sectionally (X-Y plane in figure 1) and longitudinally (Y-Z plane). The polished specimens were etched using a nital etchant (mixture of 1-ml ethanol and 10-ml nitric acid) for scanning electron microscopy (SEM) charaterizations.

Transmission electron microscopy (TEM) was performed using a Phillips CM30 microscope operating at 300 kV. The TEM samples were prepared by mechanical grinding of the twisted and untwisted Fe wires cross-sectionally and longitudinally to a thickness of about 10 µm. Further thinning to a thickness of electron transparency was carried out Gatan Precision Ion Milling System with an Ar+ accelerating voltage of 4 kV.

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EXPERIMENTAL RESULTS

The engineering stress-strain curves of the UFG and CG samples are shown in figure 2. The UFG sample has a much higher strength than its CG counterpart, as listed in Table I. The tensile yield strength (σ_{ys}) and the ultimate strength (σ_{us}) of the UFG sample are 690 MPa and 750 MPa, respectively, which are about 130 % and 103 % higher than those of the CG sample (300 MPa and 370 MPa). The elongation to failure of the UFG sample (2.5 %) is much smaller than that of the CG sample (15.8 %).

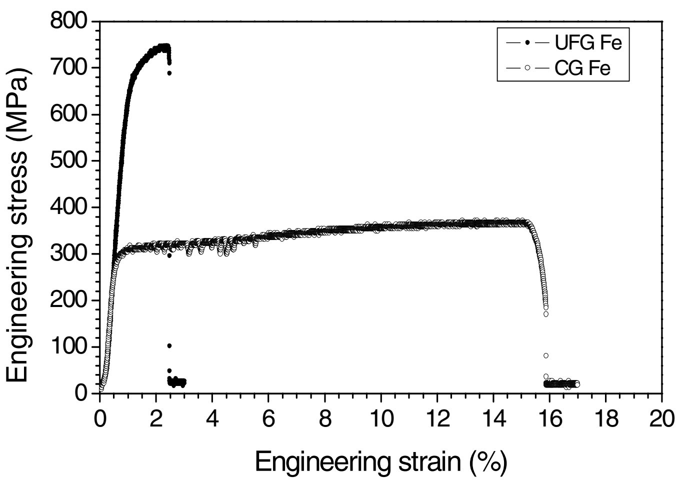

Figure 2. The engineering stress-strain curves of the twisted UFG and initial CG Fe samples.

Table I. The tensile yield strength (σ_{ys}), ultimate strength (σ_{us}) and ultimate elongation (ε_{ue}) of the twisted UFG and initial CG Fe wires.

Samples	UFG	CG
σ_{ys} (MPa)	690	300
σ_{us} (MPa)	750	370
ε_{ue} (%)	2.5	15.8

Figures 3(a-d) show the SEM pictures of the UFG and CG Fe wires. Figures 3(a) and 3(c) were taken from the cross-sectional plane (X-Y), and figures 3(b) and 3(d) were taken from the longitudinal plane (Y-Z). The CG Fe sample was composed of equi-axed grains (with an average size of about 15 μm) in X-Y plane and lamella grains (with an average size of about 25 μm) in Y-Z plane, as shown in figures 3(a) and 3(b). The micro-holes are carbide. After twist, in the cross-sectional X-Y plane, concentrically deformed layers with a thickness of about several micrometers formed surrounding the center axis of the wire, as shown in figure 3(c). The crystallites at the outer part were elongated (see positions XY-A and XY-B in figure 3c), while the grains at the center of the Fe wire remains unchanged (see position XY-C in figure 3c). In the longitudinal Y-Z plane, deformed bands with a width of about several micrometers formed, as shown in figure 3(d).

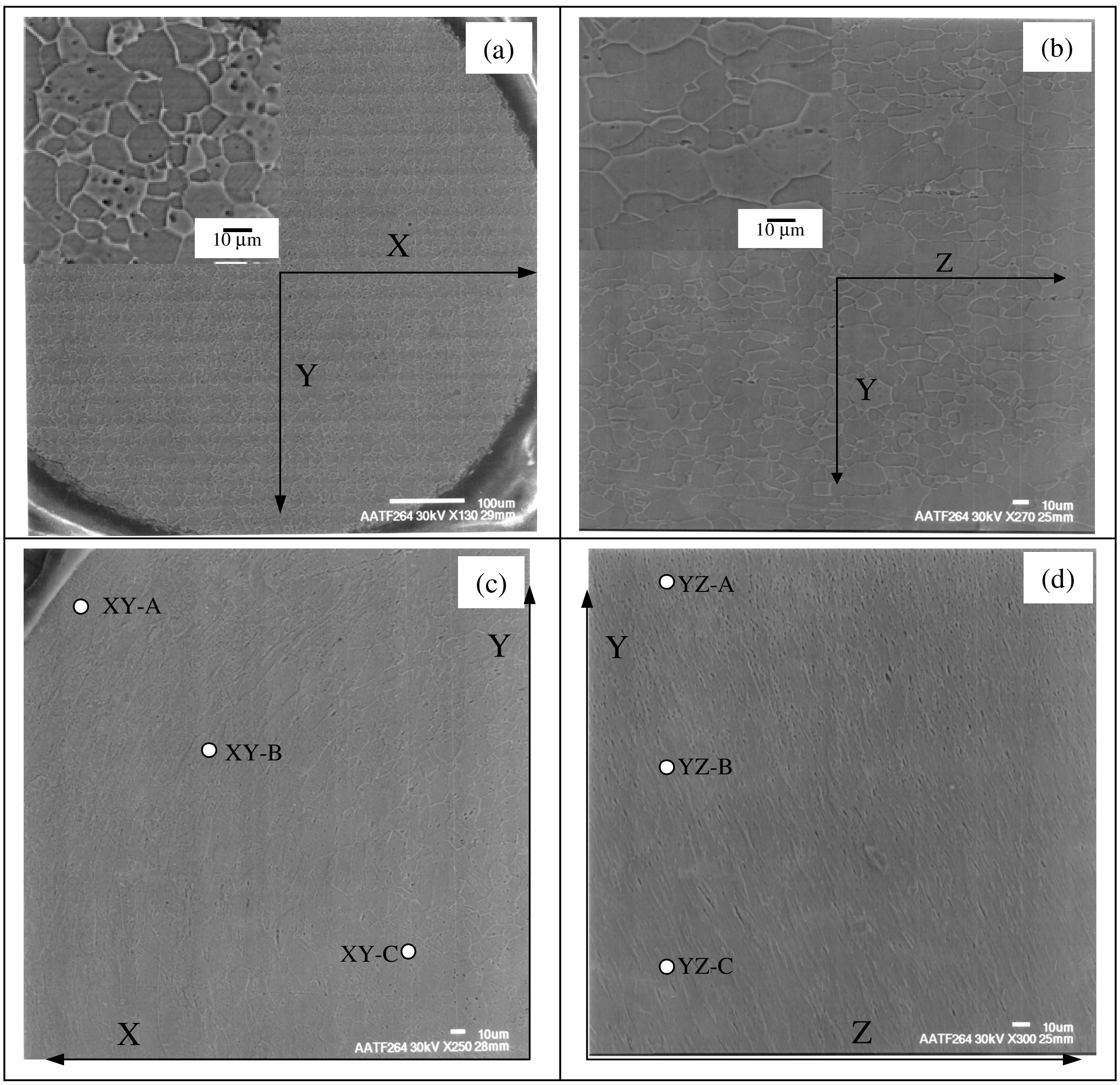

Figure 3. SEM pictures of the CG (a,b) and UFG (c,d) samples. Figures 3(a) and 3(c) were taken from the cross-sectional X-Y plane, and figures 3(b) and 3(d) were taken from the longitudinal Y-Z plane.

To study the detailed structures within the deformed layers and bands of the twisted UFG Fe wire, TEM was used to observe different positions (such as XY-A, XY-B, YZ-A, YZ-B and YZ-C in figures 3c and 3d) in the UFG sample. Figures 4(a-d) show the TEM pictures taken from positions XY-A, XY-B, YZ-A and YZ-C, respectively. One can see that near the surface position XY-A in X-Y plane, the deformed layers were composed of lamella grains with a width of about several hundred nanometers. The longitudinal direction of the lamella grains is parallel to that of

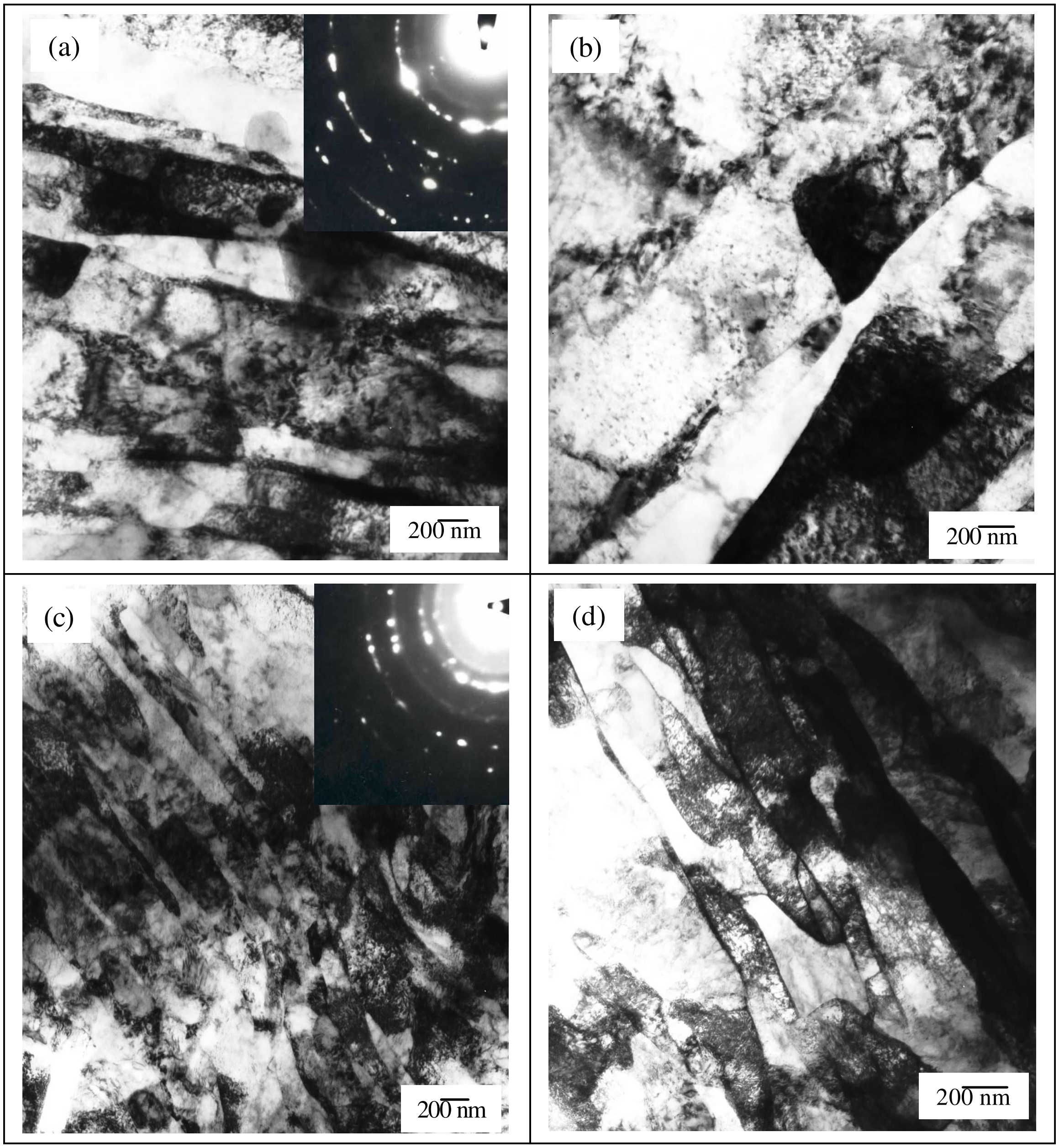

Figure 4. TEM pictures of the UFG Fe sample. Figures 4(a) and 4(b) were taken from positions XY-A and XY-B, respectively, and figures 4(c) and 4(d) from positions YZ-A and YZ-C, respectively.

the deformed layers. The deformed layers in position XY-B contain the evidently larger lamella grains than those in XY-A position, as shown in figure 4(b). The selected area diffraction (SAD) indicates the crystallographic orientations of the crystallites within the deformed layers are mainly small-angle grain boundaries, as shown the inset in figure 4(a).

From figures 4(c) and 4(d), the deformed bands in positions YZ-A and YZ-C contain lamella grains with a width of about 100-200 nm. The longitudinal direction of the lamella grains is parallel to that of the deformed bands, which has a certain angle with Z-axis. The crystallographic orientations of the crystallites within the deformed bands are mainly small-angle grain boundaries (see the inset in figure 4c).

From the TEM pictures, there are many networks within the crystallites, which are corresponding to the high density of dislocations. Moreover, the grain boundaries of the twisted sample are straight and sharp, different from the high-energy non-equilibrium boundary configurations observed in the ECAP processed UFG materials [10, 11]. This difference was caused by the different deformation mechanisms of the different SPD methods.

Figures 5(a,b) show the morphologies of the surface fracture for the tensile UFG and CG Fe samples. The fracture surface of the CG sample contains the spherical dimples, of which the size ranges from several micrometers to about 10 micrometers. For the UFG sample (see figure 5b), the fracture surface contains the concentrical-circle layers (with a thickness of several micrometers) caused by the deformed layers. Within the concentrical layers (see the inset in figure 5b), there observed the elongated dimples with a width size of about 1 μm, which were caused by the submicrometer grains within the deformed layers.

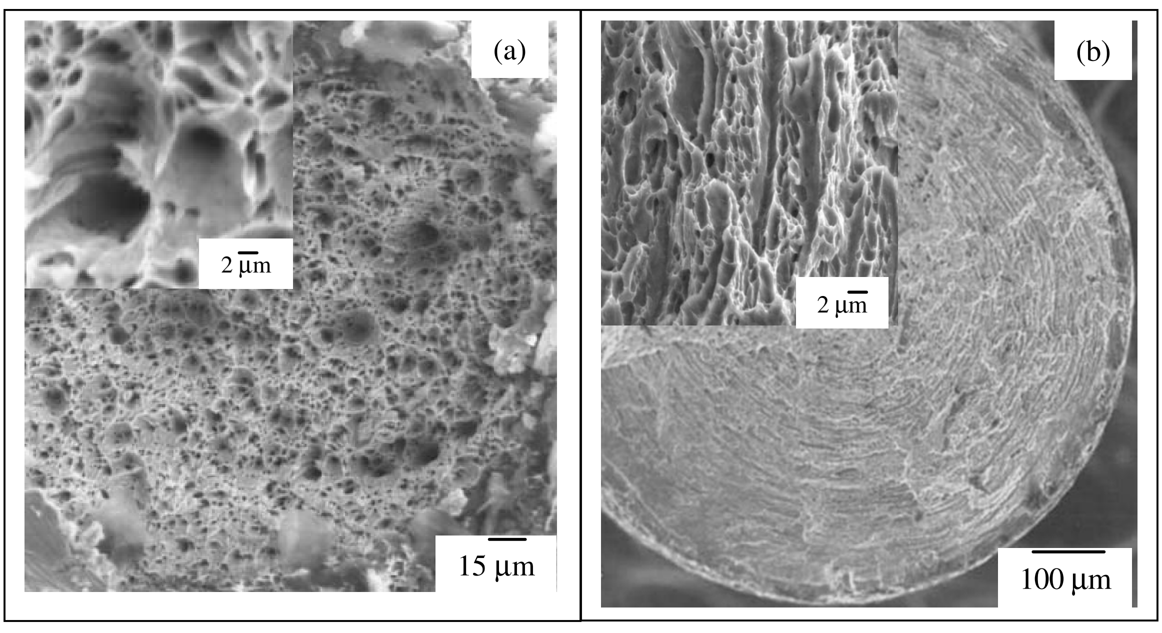

Figure 5. Morphologies of the fracture surface for the CG (a) and UFG (b) samples.

DISCUSSION

Based on above results, the twist process introduced unique UFG structures into the Fe wire. Upon twist, the deformed layers and bands with sizes of several micrometers were formed in the X-Y and Y-Z planes, respectively. Both the deformed layers and bands contain lamella grains with a width in submicrometer range. Near the surface of the Fe wire, the coarse crystallites were refined into plate shaped grains with a thickness of about 100-200 nm along Z-axis and a length of several hundred nanometers along X(Y)-axis. At the center of the Fe wire, the crystallites are coarse grained in X-Y plane, but are about 100-200 nm thick along Z-axis.

The mechanical strength of the twisted sample was enhanced by about 100 % compared with the untwisted sample. The strength enhancement was mainly caused by grain refinement strengthening and dislocation strengthening. The grain refinement strengthening is generally described by a Hall-Petch equation [12,13]. The dislocation networks and tangles within grains (as shown in figures 4a-d) will improve the resistance to dislocation movements when the moving dislocations are forced to cut through these regions of high dislocation density.

Compared to the other SPD methods, such as ECAP and HPT, the unconfined twist is simple and easy to operate. However, the processed sample must have enough plasticity to be deformed into UFG structures before fracture. For the metals and alloys lacking of enough plasticity, the twist process can be performed at an elevated temperature.

CONCLUSION

A new simple method – unconfined twist was found to effectively introduce ultrafine grained structures into the CG Fe wire. Upon twist, the deformed layers and bands were formed in the cross-sectional plane and the longitudinal plane, respectively. Both the near-surface deformed layers and the deformed bands are composed of lamella grains with a width in submicrometer range. The tensile yield strength and ultimate strength of the twisted sample are increased by about 150 % and 100 % compared with the values of its CG unprocessed counterpart. The unconfined twist SPD method shows a good prospect for industrial application.

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