ON PREPARATION OF LOW CARBON STEELS WITH NANO-GRAINED STRUCTURE AND THEIR MECHANICAL PROPERTIES

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It was found that there is few literature concerning the fining of ferrite grains by the process of severe plastic deformation and recrystallization of lath martensite, probably because both the strengthness and brittleness of martensite are very high. In this paper, the grain sizes in a high strength steel, 15CDV6, and a plain low carbon steel, Q235 were firstly fined to about 0.3μm by martensitic transfor-mation. And then, the lath martensites were aged to reduce their brittleness. Subsequently, the aged lath martensites were subjected to severe plastic deformation by cold-rolling with relative reduction of 55% ~ 93%, such a treatment resulted in the average thickness of martensite laths below 0.1μm. Before recrystallization treatment, the severe plastic deformed martensites were aged again to make the solute atoms partly segregated to the interface areas between laths and to the dislocations inside laths so as to increase the nucleating center density of recrystallization and to hinder the nuclei of recrystallization from growing. By above processing, the nanocrystalline Q235 steel sheets and 15CDV6 steel plates were prepared. The examination results of X-ray diffraction(XRD), transmission electron microscopy(TEM), tensile and microhardness tests at ambient temperature indicated that, the average grain sizes of 15CDV6 and Q235 steels were in the range of 17.4 ~ 50.6 nm. And the grain sizes decreased with the increasing of cold-rolled reduction and the decreasing of martensite lath thickness. The recrystallization temperature of 15CDV6 and Q235 steels were about 580°C and 300°C, respectively. The yield strength of 15CDV6 steel with 42nm grain size was 1360Mpa, compared with that of martensite with lath thickness of 0.3μm, increased by 190Mpa. The microhardness of Q235 steel with grain sizes of 17.4 ~ 50.6nm was 643 to532Hv, increased by 141 to 318Hv.

1. Introduction

How to refine ferrite grain size has been one of interesting and important projects in materials science and engineering field for many years[1-3]. Extensive investigations have been done in this area because the materials with ultra fine grains, especialy nano-grained structure, are expected to have unusual properties, such as high strength and ductility, superplasticity, damping, negative coefficent to the Hall-Petch relation and changes of usually structure-insensitive parameters, e.g. elastic moduli, Curie and Debye temperature, saturation magnetization,etc [2,4,5].

There are many techniques for minimising grain size, such as alloying,controlled rolling combined with accelerated cooling, plastic deformation and recrystallization, gas condensation with subsequent consolidation, ball-milling, amorphous crystallization, equal-channel angular pressing, strain-induced trans-formation from austenite to ferrite, etc [2,3,5-8]. Among these methods, severe plastic deformation is a very attractive method for it having a number of advantages. First, massive samples (sheets, rods) can be produced, which is very useful for mechnical testing and physicochemical examination. Secondly, no residual porosity is found in the sample produced. Thirdly, it is possible to reveal the intrinsic properties of nanocrystalline materials.

The conventional plastic deformation and recrystallization (PD&R) used in indusry and academic studies for refining are mainly focus on soft materials, such as non-ferrous metals, their alloys and annealed steels[6-8]. However, the fining effect of PD&R applied to annealed structure in steels is very limited because the annealed microstructure is very coarse and heterogeneous. It is well known that the refined grain size as small as sub-micrometer, even nano-scale can be obtained through martensite transformation[9], but the grain shape of martensite is unequiaxial. If a low carbon steel with lath martensite microstucture were subjected to severe plastic deformation to reduce the lath thickness below 0.1μm, subsequently had it recrystallized, these laths would probally become nanocrystalline ferrite with equiaxial grains [10].

Based on above, in the preset work, low carbon steels were used to explore the feasibility of the proparation of nanocrystalline steel sheets by a combination of martensitic trans-formation, severe plastic deformation and recrystallization of lath martensite.

2.Experimental Procedure

The steels used in this work were a high strength steel, 15CDV6, and a plain low carbon steel, Q235. Their chemical compositions were listed in Table 1. A schedule of heat treatments and plastic deformation used to obtain nano-grained structure for each steel is given in Table 2.

The room temperature measurements of the microhardness(Hv) for Q235 samples were performed on a microhardness tester using a Vickers diamond pyramidal indenter with a load of 1.96N and the duration of testing was 10S. The tensile properties of 15CDV6 samples were studied by ambient temperature uniaxial tensile testing in a universal testing machine of the Instron type, with a constant strain rate of 10^{-4} S^{-1}.

Both the microstructure and the grain size of these two steels were characterized using optical microscopy(OM), transmission electron microscopy (TEM) and X-ray diffraction(XRD) techniques. The TEM studies were conducted on a H800 electron microscope. For TEM examination, blanks were cut from the steel sheets and mechanically thinned to about 0.05mm. These foils were then jet polished at a temperature of -20°C on a twin jet polishing apparatus operated at approximately 75 to 85 volts and 40mA DC, in an electrolyte compring 8% perchloric acid, 15% methanol and 77% acetic acid.

Table 1

Chemical Composition (Wt Pct)

	C	Mn	Si	Cr	Mo	V	S	P
15CDV6	0.16	1.01	0.18	1.28	0.86	0.26	0.014	0.018
Q 235	0.17	0.68	0.37	---	---	---	0.039	0.036

Table 2

Thermomechanical Treatment Summary

Materials	Thermomechanical Treatment
15CDV6	Austenized 25 min at 940°C, Oil quenched Aged 90min at 550°C Cold rolled with relative reduction of 55% ~75% without any intermediate annealing Aged 90min at 580°C Recrystallized 60 to 1440min at 550°C to 700 °C
Q235	Austenized 25 min at 880°C Aged 90 min at 180°C Cold rolled with relative reduction of 93% without any intermediate annealing Aged at room temperature to 500°C for 30 to 120min

3.Results and Analysis

3.1.Microstructure and Grain size

The microstructures of cold-rolled in 15CDV6 and Q235 steels were shown in Fig.1 a and b, respectively. Compared with the lath martensite before rolled, the average thickness of laths reduced from about 300nm to 90nm in 15CDV6 steel. Inside the severe plastic deformed laths, the dislocation density was very high, and no carbide was observed. In Q235 steel, however, there were many very small equiaxied grains with high density dislocations instead of deformed laths, as shown in Fig.1 b. This implies that dymamical recrystallization of severe deformed martensitic laths occurred during cold-rolling.

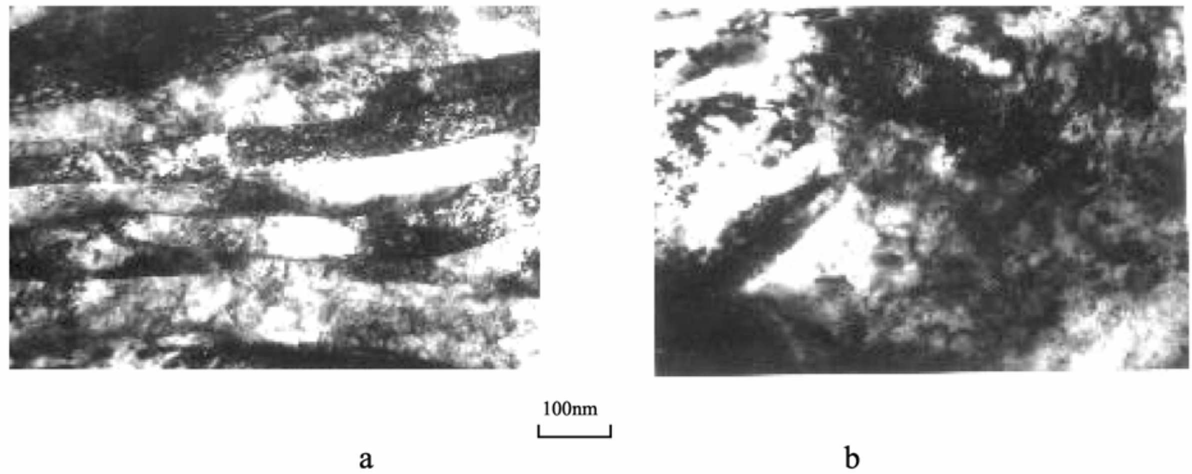


Fig.1. TEM images of cold-rolled lath martensite in 15CDV6 steel (a) and Q235 steel (b)

The microstructure of cold-rolled and recrystallized samples of 15CDV6 and Q235 were shown in Fig.2 a and b, respectively. Fig.2 indicaed that the general feature of all microstructures in both 15CDV6 and Q235 was the presence of equiaxed ferrite grains with grain size of 20 to150nm.

In order to determine the grain size, TEM dark field image technique and XRD scherrer method were conducted. For each sample, at least 250 grains in TEM dark field image were measured; the results were given in Table 3.

Table 3

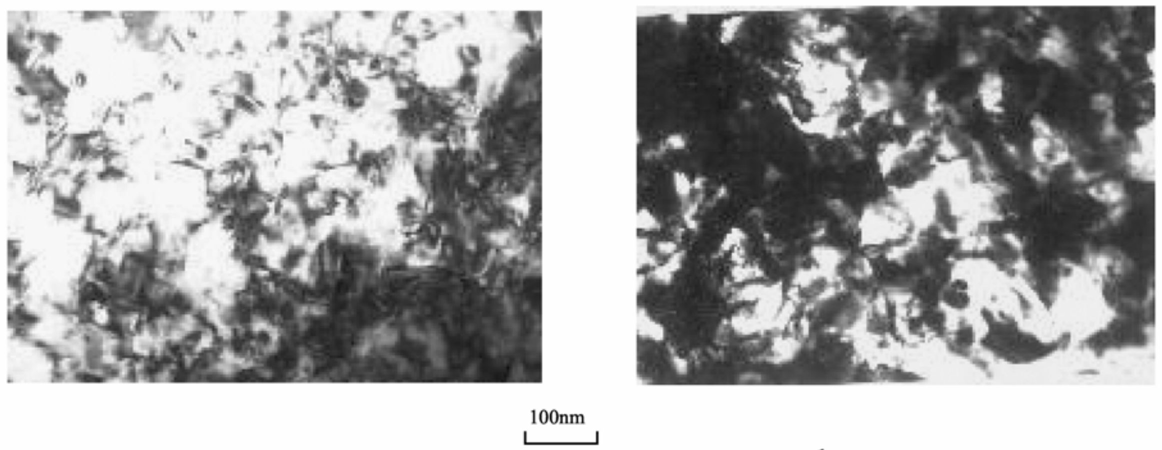
Grain sizes nm determined by TEM dark field images and XRD scherrer method

		011	200	211	average	processing
XRD	15CDV6	51.8	32.4	35.4	39.9	75% reduction at 580°C for 16h
	Q235	20.0	11.9	13.9	17.4	93% reduction at room tempera-ture
		23.1	16.6	14.8	18.2	300°C x60min
		28.9	13.8	17.5	20.1	350°C x60min
		72.3	34.5	43.7	50.2	400°C x60min
TEM	15CDV6				46.0	75% reduction at 580°C for 16h
	Q235				18.9	At room temperature 93% reduction
					51.8	400°C x60min

3.2. Mechanical properties

Tensile properties indicated that very high strength combined with good ductility were achievable with this type of processing, as shown in Table 4. The yield strength (YS) of 15CDV6 under conventional processing was about 1100Mpa, whereas the same steel consisting of an Ultra-fine ferrite microstructure displayed YS of between 1320 and 1467Mpa. Compared with that of lath martensite, both YS and TS increased by about 190Mpa. In addition, ductility remained acceptable in most cases with values of total elongation of about 16% and values of area reduction of about 50%.

The effects of recrystallization parameters, such as temperature and time, on the microhardness of Q235 steel with 93% reduction were summarized in Table 5. Before cold rolling, the microhardness of Q235 steel with lath martensite microstructure was 382Hv.



a b

Fig.2. TEM images of recrystallized microstructures in 15CDV6 (a) and in Q235 (b) and (c) 75% reduction, at 580°C for 16h. (b) 93% reduction, at 300°C for 1h

Table 4

Tensile properties of 15CDV6 steel under different processing

Roll reduction	Recrystallization	YS (Mpa)	TS (Mpa)	YS/TS	δ(%)	ψ(%)	grain size (nm)
55%	580°C x16h	1320	1408	0.94	16.6	52.0	76
65%	580°C x16h	1346	1423	0.95	16.2	51.0	58
75%	580°C x16h	1467	1502	0.97	15.8	49.0	42
	940°C quenched 550°C x6.5h	1170	1226	0.90	18.5	55.9	300

Table 5

The relationship between microhardness and processing parameters in Q235 steel.

Temperature (°C)	Recrystallization time (min) 20	40	50	60
300°C	643	632	569	532
400°C	---	---	---	446
500°C	---	---	---	328
600°C	---	---	---	191

* Before cold rolling, the microhardness of Q235 steel with lath martensite microstructure was 382Hv

A noteworthy phenomenon is that the severe plastic deformed martensitic laths were dynamically recrystallized during cold-rolling, and transformed into very tiny grains. The observations of both TEM and XRD, see Fig.1 b and Table 3, revealed that, after cold-rolling the grain size was about 17~20nm, the corresponding microhardness was as high as 640Hv, compared with that of lath martensite before rolled, increased by 318Hv. The samples with grain size of 18.2nm had values of 532Hv, increased by 150Hv.

4. Conclusions

4.1 Nanocrystalline steel sheets of 15CDV6 and Q235 were prepared by a combination process of quenching, aging,heavy cold rolling and recrystallizing.

4.2 An interesting phenomenon found in this work is the dynamical recrystalization of severe deformed lath martensite occurred during cold rolling procedure, this results in a micro-structure with very tiny grains of about 20nm and very high hardness level of 640Hv.

4.3 Contrary to Q235 steel, the thermostability of the severe deformed lath martensite in 15CDV6 steel is very high, recrystallization finished at 580°C took 16 hours.

Acknowledgement

This work was partially funded by the National Nature Science Foundation of P.R.China under grant No.59681006 and the Nature Science Foundation of Hebei Province under grant No.595165.

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Article is delivered in editing 9.07.2002