Austenite Stability and Its Effect on the Ductility of the Cold-rolled Medium-Mn Steel

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The mechanical and thermal stability of retained austenite in the cold-rolled medium-Mn (Fe-0.1C-5Mn) steel were studied by the tensile deformation at different temperature. The volume fractions of retained austenite and microstructure of the starting and deformed materials were analyzed by X-ray diffraction, the scanning electron microscopy (SEM) and electron back scattered diffraction (EBSD). It was found that the volume fraction of retained austenite gradually decreased with strain increasing during tensile deformation. It showed high mechanical stability of retained austenite, which resulted in good comprehensive mechanical properties with ultrahigh strength and plasticity. Meanwhile, the tensile results at different temperature showed pretty high plasticity at low temperature (−40°C and −80°C) tensile deformation of the cold-rolled steel, which indicated the retained austenite maintained certain stability at low temperature. The results of this study indicate that the ductility in the medium-Mn steel with high contents of retained austenite can be altered by control of the austenite stability.

KEY WORDS: cold-rolled medium-Mn steel; mechanical and thermal stability; retained austenite; microstructure; ultrahigh strength and plasticity.

1. Introduction

The TRIP (transformation induced plasticity) steels have been studied for over 40 years.^{1,2)} The excellent combination of the high strength and plasticity of these steels was attributed to the strain-induced austenite-to-martensite phase transformation during plastic straining. During the past few decades, there was a rapid development in the automobile industry in order to meet the demand for energy reduction and safety improvement. Since the TRIP effect contributes to the high work-hardening rate of the microstructure and delays the onset of necking,³⁾ they have been received increasing attention as new high-formability cold-rolled steel sheets for automobile bodies.^{4–7)}

Nowadays the studies on the development of a new generation of steel with high strength and high ductility at reasonable cost have been promoted worldwide. In light of the high ductility contributed from metastable austenite in TRIP steel and stable austenite in TWIP steel,^{8–10)} together with the high strength from grain refinement by severe plastic-deformed materials,¹¹⁾ increasing the volume fraction of retained austenite and refining the grain size into the sub-micron region in high-strength low-alloyed steel is a promising approach to design a new type of steel with both ultrahigh

strength and high ductility.¹²⁾ Previous studies^{13–15)} confirmed that the volume fraction, the distribution of various phases, especially the stability of retained austenite is of great importance to the mechanical properties of transformation induced plasticity (TRIP) steels. However the stability of the retained austenite could be effectively varied by deformation temperature.¹⁶⁾ Several studies^{17–20)} on the relationship between deformation temperature, mechanical properties, and the stability of retained austenite have been performed. Since 2007, the medium-Mn steel, one of the typical third generation automobile steel, with ultrafine grain size and large fractioned austenite was reported with high strength and high ductility.²¹⁾ However seldom experiments were demonstrated on its deformation behaviors at very low temperature to show mechanical property dependence on the deformation temperature.

In this study, the cold-rolled medium-Mn steel was processed by long time annealing in the intercritical region. The mechanical properties were measured by the tensile test at different temperature. Microstructure evolutions during annealing and tensile deformation were studied by SEM, EBSD and XRD. The mechanical stability of retained austenite was calculated by the volume fraction of retained austenite within different tensile strain. The relationship between the ductility and the austenite volume fraction was analyzed tentatively.

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2. Experiment

In this study the cold-rolled medium-Mn steel was fabricated and applied to study the stability of retained austenite during the deformation. The chemical composition of the test steel investigated in the present work was 0.1 wt% C and 5 wt% Mn. The vacuum melted material was first made as a hot rolled strip with a thickness of 4 mm through the conventional hot rolling process. Then, the strip was cold rolled to a thickness of 1.2 mm. In order to obtain the TRIP behavior, the cold rolled strip was further processed by austenite reverted annealing at 650°C for 18 h air-cooled to room temperature.

In order to study the mechanical stability of the retained austenite, the steel was deformed by tensile test at room temperature. Then the steels were machined into a tensile specimen according to the standard geometry of the tensile specimen (GB/T 228.1-2010). The uniaxial tensile test was carried out with strain rate of 2.5×10^{-4} /s on the WE300B tensile testing machine. The mechanical properties were measured from the test machine. For the measurements of the retained austenite volume fraction, the tensile specimens with different tensile strain were made. Finally the tensile tests were conducted at temperature of -196°C , -120°C , -80°C , -40°C , 25°C and $+100^\circ\text{C}$.

The X-ray diffraction was used to determine the retained austenite volume fraction on the rolling surface after grinding and polishing. After mechanical polishing the rolling surface was electrolyzed for about 50 s with 10% chromic acid electrolysis (voltage 5V) to remove the surface stress, then the residual austenite content was determined by PHILIPS APD-10 X-ray diffraction measurements. The Co target was used in the X-ray diffraction with tube current of 30 mA and voltage of 30 kV. The graphite crystal monochromatic was used get to the diffraction patterns with the step scan mode in which scanning 2θ ranged $45\text{--}115^\circ$, step was 0.02° and integration time was 0.4 s.

For EBSD observation, the specimens were first mechanical polishing and then electro-polished at room temperature in a solution of 90% ethanol and 10% perchloric acid solution. EBSD scans were performed at 20 kV with a step size of $0.05\text{ }\mu\text{m}$ and the measured map sizes are $50 \times 40\text{ }\mu\text{m}^2$. In order to measure the microstructure, the EBSD maps were post-processed by reducing all the single pixels, which were considered to be the wrong indexed results. Due to the misorientation resolution limitation of EBSD, only the grain boundaries with misorientation angle larger than 2° were

given in the EBSD maps.²²⁾ The samples for SEM were treated by means of cold grinding and polishing, corrosion for 15 s with 3% nitric acid alcohol solution, and microstructure observation was conducted in S-4300 cold field emission scanning electron microscopy (SEM). The stress strain curves at different temperature were analyzed to understand the work hardening mechanism of steel during deformation process.

3. Experimental Results

3.1. Microstructure Characterized by SEM

The microstructure of the annealed steel was characterized by the SEM as shown in Fig. 1. It can be seen that the microstructure mainly consisted of the convex and concave grains. As reported in precious studies,²¹⁾ the austenite reverted transformation took place during the annealing process in the medium-Mn steel, so the microstructure was supposed to be the mixture of the slightly recovered ferrite grains and a few of austenite grains. As the austenite grain was easy to be etched and the ferrite grain was difficult to be etched, the concave grains were identified as the austenite grains. The average grain size of the recovered ferrite phase was about $1.5\text{ }\mu\text{m}$. Moreover, there was a bit of carbides located on the surface of ferrite phase. An outstanding different structure was also found in Fig. 1(a). The magnified SEM map was shown in Fig. 1(b) with the energy spectrum analysis. From Fig. 1(b), the structure was something like lamellar pearlite. And from Fig. 1(b), the C element (red line) and Mn element (green line) was rich in this structure and showed an obvious fluctuation between the lamellar structure. Therefore this structure was supposed to be the lamellar pearlite which may be formed during the sluggish cooling from the austenite.

The microstructures of the medium-Mn steels deformed at different tensile strain were characterized by the SEM as shown in Fig. 2. It can be seen that the microstructure was quite similar as the anneal state and the lamellar typed pearlite phase was a very obvious phase, which was never demonstrated in previous study. The volume fraction of retained austenite (concave phase) decreased with strain increasing. From Figs. 2(a) to 2(d), it can be seen that the slip lines

Fig. 1. Microstructures of the long time annealed cold rolled medium-Mn steel. (a) the microstructure of the annealed medium-Mn steel with ferrite grains, austenite grains and some pearlite phase, and (b) the chemical composition analysis by energy spectrum analysis in SEM.

Fig. 2. Microstructure of tensile deformed steel at room temperature characterized by SEM, (a) 2% elongation, (b) 7% elongation, (c) 20% elongation and (d) 30% elongation.

could be clearly found in the large sized ferrite grains, which is much predominated in the steel after 30% tensile deformation. The grain size of the ferrite is range from $0.25\ \mu\text{m}$ to $2.5\ \mu\text{m}$ with average grain size about $1.0\ \mu\text{m}$, and the grain size of the austenite is only about $0.25\text{--}1.0\ \mu\text{m}$ with average grain size about $0.5\ \mu\text{m}$. However, the size of the pearlite colony is about $1.0\text{--}1.5\ \mu\text{m}$ with average size of about $1.25\ \mu\text{m}$.

3.2. Microstructure Characterized by EBSD

The microstructure of the deformed steels was characterized by EBSD in normal rolling plane taken from the tensile samples which was deformed to different strain as shown in Figs. 3(a) and 3(b). In these figures the retained austenite grains are red colored, the ferrite and pearlite are white colored. The high angle boundaries and low angle boundaries are depicted as black lines and green lines, respectively. It can be seen from this figure, the starting macrostructure is a duplex phase structure including both ferrite and austenite since it is very difficult to discern ferrite phase and pearlite phase due to the nearly same lattice parameter between both

Fig. 3. The deformed microstructure of steel deformed at room temperature determined by EBSD, (a) 2% elongation; (b) 20% elongation. In which low-angle boundaries ($2^\circ \leq \theta \leq 15^\circ$) and high-angle boundaries ($\theta > 15^\circ$) are depicted by green lines and black lines, respectively. The red color areas represent the retained austenite.

phases.²³⁾

From Fig. 3(a), it can be seen very clearly that the grains of the retained austenite are located in the boundary regions of the ferrite and/or pearlite phases. As the retained austenite grains are red colored, the volume fraction of austenite was 6.41% calculated from the EBSD map in the sample with 2% elongation and there was only 0.04% in the sample with 20% elongation. It revealed that the volume fraction of retained austenite decreases with strain increasing. Compared Figs. 3(a) and 3(b), it can be seen that there were more low angle boundaries with the strain increased to 20%, which implied fast accumulation of dislocation due to plastic deformation and phase transformation. Even after 20% elongation, there are some areas (mainly large ferrite grains) still less deformed with no clear low angle boundary formation, which implies that the deformation behaviors of different phase are different due to the different interaction behaviors among phases and thus results in the heterogeneous deformation structure.

3.3. The Mechanical Properties

The stress-strain curves of steels which were tensile tested at different temperature are given in Fig. 4. It can be seen that the stress strain curves at 100°C and -120°C showed similar features while the behavior at -196°C was different without necking elongation, which may be due to sharp transformation from the austenite to the martensite and the rapid expansion of cracks at such low temperature. In all the cases the stress increase with strain increasing but the work hardening rate decrease with strain increasing as shown in Fig. 4(b). Also in the beginning of deformation, the work hardening rate is higher than that at higher temperature.

The variations of the yield strength, tensile strength and total elongation are plotted in Fig. 5 as a function of tensile-testing temperature. It can be seen that both the tensile strength and the yield strength (sometimes instead by the

Fig. 4. Tensile stress strain curves of the cold rolled medium-Mn steel tensile deformed at different temperature (a) engineering stress-strain curves and (b) true stress and strain curves.

Fig. 5. Mechanical properties as a function of tensile-testing temperature to show the deformation mechanism (a) strength and (b) elongation.

strength of 0.2% offset Rp0.2) decreased with increase of tensile test temperature. The decrease in the yield strength was caused principally by the effects of thermally activated plastic flow. It is very interesting that the total elongation was over 35% even the deformation temperature was -80°C , which implied that the retained austenite was still stable leading the TRIP effect to improve the plasticity.

The M_s is the temperature at which the austenite starts to form on pre-existing nucleation sites during cooling, driven by the difference in the chemical free energy $\Delta G^{\gamma \rightarrow \alpha'}$ of the two phases.²⁴⁾

The temperature region $M_s \leq T \leq M_{\sigma}$ is therefore usually referred to as the stress-assisted transformation regime, while $M_{\sigma} \leq T \leq M_{\epsilon}$ is called the strain-induced transformation regime.²⁵⁾ Based on the dependence of the yield stress and the tensile stress as shown in Fig. 5(a), the martensite start transformation temperature of the retained austenite (M_s) is about -120°C , the stress induced martensite transformation (M_{σ}) is about -40°C and the strain induced phase transformation temperature (M_{ϵ}) is about 25°C , which is in a very good agreement with the variation of the total elongation with deformation temperature as shown in Fig. 5(b).

4. Discussion

4.1. Microstructure Evolution and the Mechanical Stability of the Retained Austenite

Based on the results from EBSD as shown in Fig. 3, it can be seen that the low angle dislocation boundaries developed during deformation process and the low angle boundary density increases with strain, indicating that the microstructure refines during deformation process. It can be seen from EBSD map, even after 20% elongation, the dislocation boundaries still don't develop in some large ferrite grains. In addition, the fraction of the retained austenite decreased with the strain which resulted in the higher fraction of martensite phase due to the TRIP effect. Therefore it indicated a significant dependence of dislocation density on phase transformation. The strong dependence of dislocation density on the martensite volume fraction can be interpreted by the volume expansion during phase transformation, in which process high density dislocation around the martensite grains has been produced to accommodate the volume expansion.

It can be seen qualitatively from Fig. 2, the retained austenite fraction decreases with strain increasing during deformation process. The volume fraction of the retained

austenite in samples deformed to different strain was determined by the XRD and the results are given in Fig. 6(a). It can be seen clearly in Fig. 6(a) that the volume fraction of retained austenite gradually decreased with strain increasing. In the range where the true strain is lower than 0.11, the retained austenite fraction drops dramatically with increasing strain, indicating very fast strain-induced transformation. Even the true strain rises over 0.17; the austenite fraction of the steel still decreases very slowly, indicating that the strain-induced transformation still continues.

There are several equations which represent the relationship between an applied strain and volume fraction of resultant martensite transformation.²⁶⁾ The Eqs. (1) and (2) were used to represent the relationship between deformation strain and volume fraction of retained austenite as suggested by Chung²⁷⁾ and Sugimoto.²⁸⁾ However, in this study, the value of $f_{\gamma, u}$ is 0, which means there is no "dead retained austenite" as proposed by these authors. Thus the changing formula of retained austenite can be rewritten as in Eq. (2).

$$\frac{f_{\gamma, \epsilon} - f_{\gamma, u}}{f_{\gamma, 0} - f_{\gamma, u}} = \exp(-k\epsilon) \quad \text{..... (1)}$$

$$\frac{f_{\gamma, \epsilon}}{f_{\gamma, 0}} = \exp(-k\epsilon) \quad \text{..... (2)}$$

where, $f_{\gamma, 0}$, $f_{\gamma, u}$ and $f_{\gamma, \epsilon}$ refer to the austenite volume fraction in the starting materials, the volume fraction of retained austenite after broken during tensile and the volume fraction of retained austenite at given strain (ϵ), respectively. k is defined as the stability coefficient of retained austenite, and the larger k value indicates the lower stability of retained austenite and the faster martensitic transformation.

The fitting line between the true strain and the fraction of retained austenite in natural logarithm scale was shown in Fig. 6(b). The slope of straight line corresponded to the k value in Eq. (2). In general, the lower k value indicates higher stability of retained austenite.²⁵⁾ This stability of retained austenite is closely related with the mechanical properties of cold-rolled steel sheets, with uniform elongation in particular. This is because necking is delayed as the strain-induced transformation lasts up to the higher strain region when the stability of retained austenite is high. The mechanical stability value of k under tensile test at room temperature is 12.9, which is a little lower than the k (from 16.20 to 20.99) in the TRIP steel,¹³⁾ indicating a very high mechanical stability of the retained austenite in the cold rolled medium-Mn steel than that in the conventional TRIP steels.

Fig. 6. The volume fraction of retained austenite at different tensile strain of the steel deformed at room temperature (a) in normal scale and (b) in natural logarithm scale.

Fig. 7. The relationship between deformation temperature, calculated mechanical stability (k_T) through Eqn. (3) and the ductility (a) AT, AU and k_T as a function of the deformation temperature and (b) AT and AU as a function of the inverse of k_T .

4.2. The Thermal Stability of Retained Austenite and Its Effects on the Plasticity

As we all know the M_s is the temperature at which the austenite starts to transform to martensite on nucleation sites due to the super cooling. The different in the free energy between the two phases $\Delta G_{\gamma \rightarrow \alpha'}$ afford the drive force and energy for the transformation. At temperatures above M_s , the transformation can occur unless additional driving force U is assisted. In this study, the additional driving force was provided by the tensile test. Furthermore, as the increase of the temperature, the additional driving force increase which implied that the austenite stay more stable at higher temperature during tensile test.

Based on the Eq. (2) and above conclusion, the k is defined as the stability coefficient of retained austenite. In order to make it more generally application, it is necessary to express k as a function of the chemical composition of the austenite and of the deformation temperature. The simplest assumption is to assume that k is proportional to the driving force as proposed by M. Y. Sherif:²⁹⁾

$$f\gamma, \varepsilon = f\gamma, 0 \exp(-k\varepsilon) \quad \text{with } k = k_1 \Delta G \dots\dots\dots (3)$$

ΔG is the chemical free energy change $\Delta G = G^\gamma - G^{\alpha'}$ (to make it positive) for the transformation of austenite to ferrite and it is related to both the chemical composition and the deformation temperature. The coefficient k_1 was a constant and its value was $0.00446 \text{ molJ}^{-1}$.²⁹⁾

In order to derive chemical driving force (ΔG) of the medium-Mn steel (the chemical composition of the austenite of 0.6%C and 9%Mn was used), the commercial Thermal-Calc software was applied to calculate the relationship between ΔG and the phase transformation temperature (T), which could be written as follows,

$$\Delta G = 3339 - 14.7T \dots\dots\dots (4)$$

in which the unite of temperature (T) degree (°C).

Based on Eqns. (3) and (4), the austenite stability at room temperature could be calculated to be 13.25, which is in a very good agreement with our experimental value of 12.9. Then the austenite stability at different temperature was calculated based on Eqns. (3) and (4). The calculated k and measured A_T and A_U are given in Fig. 7(a) and the dependence of A_T and A_U on the mechanical stability are shown in Fig. 7(b). It can be seen clearly that the uniform elongation (A_U) is in a good linear relationship with the inverse of k_T in semi-log scale, but total elongation (A_T) is not strongly dependent on it. This means that the uniform elongation is controlled by the TRIP effects of the retained aus-

tenite and can be described by the linear relationship as shown in Fig. 7(b). Further study on this dependence should be carried out in near future. Thus even though it was usually said that the total elongation to failure of TRIP-aided steel is mainly controlled by the strain-induced transformation of retained austenite³⁰⁾ during the tensile test since the martensitic transformation in TRIP steel has “strain hardening” effect and “uniform elongation” effect due to redistribution of stresses. Actually only the uniform is meaningful to the TRIP effects of the retained austenite as shown in Fig. 7(b). Figure 7(b) also tells us that the stability of retained austenite is an important factor directly affecting ductility of the TRIP steels.

As the results shown above, it revealed a very good comprehensive mechanical property with the tensile strength 700 MPa and the total elongation 40% in the test steel at room temperature. Moreover the product of the tensile strength to the total elongation was 28 GPa*%, which is higher than that of the conventional TRIP steel. Meanwhile the stability coefficient of retained austenite was calculated to be 12.9 which indicated that the austenite was much more stable than that of the conventional TRIP steels during tensile test. Therefore it can be inferred that the rather high stability of the retained austenite ensured the pretty high elongation of the medium-Mn steel as necking is delayed as the strain-induced transformation lasts up to the higher strain region, which results in a very high uniform elongation. In addition, it is very interesting that the engineering stress-strain curves of samples tensile tested at -40°C and -80°C are quite similar with that of sample deformed at room temperature. Moreover the total elongation was over 35% even the tensile test was conducted at -80°C since at low temperature austenite is less stable against straining and transforms into martensite very rapidly. As mentioned above, the high elongation of the steel confirmed that the austenite was stable enough at low temperature (-40°C and -80°C) to ensure its high ductility during the tensile test.

5. Conclusions

In this study, the mechanical and thermal stability of the cold-rolled medium-Mn steel was investigated. The microstructure was characterized by SEM and EBSD and the tensile deformation with different strain at room temperature and tensile tests at different temperature were conducted respectively. The main conclusions of this study are summarized as follows,

- (1) It was demonstrated that the recovery and austenite

reverted transformation took place during the annealing process in the cold-rolled medium-Mn steel. The ferrite and austenite microstructure was obtained in the steel in addition some lamellar structure pearlite was also found, which may be formed during the sluggish cooling from the austenite.

(2) It revealed a very good comprehensive mechanical property with the tensile strength 700 MPa and the total elongation 40% in the steel deformed at room temperature. At the same time the stability coefficient of retained austenite k was 12.9 indicated that the retained austenite was rather stable during the tensile test, which ensure the high strength and high plasticity was obtained in the test steel.

(3) The tensile test of the medium-Mn steel at low temperature revealed rather high plasticity which indicated that the austenite can keep stable at low temperature tensile test. Therefore the cold-rolled medium-Mn steel is expected to be applied in some occasions which are exposed to the low temperature.

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