Effect of Nb and Cu on the microstructure and hardness of quenching–partitioning–tempering dual-phase steels

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ABSTRACT
The effect of Nb and Cu on the microstructure and hardness of quenching–partitioning–tempering (Q–P–T) dual-phase steels was investigated. Nanostructured martensite and retained austenite were obtained when the steels were treated with a Q–P–T process. The hardness, volume fraction of retained austenite and final carbon content in austenite of the Nb + Cu-containing steel were higher than the Nb-containing steel. This was attributed to the promotion of Nb precipitation by the addition of Cu.

Materials and experimental procedures
The chemical composition of the newly designed alloy 1 and alloy 2 is listed in Table 1. To produce the designed alloys, high purity materials were used and melted in a vacuum induction furnace, and then rolled into bars with the dimension of 50 mm × 30 mm. The homogenization was performed in a vacuum furnace at 1150°C for 48 h. During the Q–P–T treatment, specimens were firstly austenitized at 870°C for 30 min, and immediately quenched into the molten salt bath at 210°C; and then partitioned at 450°C in the molten salt bath for 60, 120, 600 and 1800 s, subsequently quenched into room temperature water.

The microstructure characterization was carried out with optical microscope (OM), scanning electron
microscope (SEM) and transmission electron microscope (TEM). The specimens for OM and SEM were ground and polished using standardized techniques and etched in 4 vol. % nital solutions after heat treatment. Selected specimens for TEM observations were thinned to 0.3 mm with wire cutting and abraded to 0.06 mm with SiC papers. Then the 3 mm punched disk was twin-jet electropolished using 5% perchloric acid solution to obtain enough thin areas for microstructure observations.

Quantitative X-ray analysis (XRD) was used to determine the volume fraction of retained austenite and the amount of retained austenite. The samples were step scanned in an X-ray diffractometer, operating at 40 kV voltage and 45 mA current, using Cu Kα radiation. The 2θ scan angles ranged from 30° to 100° with a scanning speed (2θ) of 5°/min. The calculations were based on the integrated intensities of (200) α, (211) α, (200) γ, (220) γ and (311) γ diffraction peaks [9]. The carbon concentration in austenite was estimated using the measured lattice parameters [10].

Vickers hardness tests were reported as the average of at least 10 tests. The tests were conducted using a 1 kg load, and each value represents the mean of those measurements.

### Table 1. Chemical composition of the designed steels (wt. %)

| Alloy | C   | Si  | Mn  | Nb  | Cu | Fe  |
|-------|-----|-----|-----|-----|----|-----|
| 1     | 0.56| 0.68| 1.52| 0.21| –  | Bal.|
| 2     | 0.55| 0.75| 1.61| 0.22| 1.08| Bal.|

**Results and discussion**

Typical OM and SEM micrographs of alloy 1 and alloy 2 quenched at 210°C, then partitioned at 450°C for 600 s are shown in Figure 1. It clearly revealed the morphology of tempered martensite and retained austenite. It can be seen that most of the retained austenite with thin film-like and blocky morphology was maintained after partitioning.

Figure 2 shows the TEM images for the Nb-containing and Nb + Cu-containing steels treated with the process of quenching at 210°C and partitioning-tempering at 450°C for 1800 s. It is noted that the average thickness of martensite was 450 ± 30 nm and 400 ± 25 nm for Nb-containing and Nb + Cu-containing steel, respectively. It can be seen that the microstructure of Nb + Cu-containing steel was finer than that of Nb-containing steel. Figure 3 presents the TEM micrograph of precipitates by means of carbon extraction replica in the specimens of Nb-containing and Nb + Cu-containing steels, which were treated by the process of quenching at 210°C and partitioning-tempering at 450°C for 120 s. It is seen that there were large numbers of fine carbides dispersed in the matrix, and these carbides appeared to be in equiaxial morphology with an average size of 20 ± 5 nm. With the addition of Cu, the average thickness of martensite was reduced and the size and number of NbC precipitates were increased. This means that the adding of Cu can speed up the precipitation. The fine particles of Nb and Cu precipitates will pin the interface boundary and
hinder the coarsening of martensite laths, and thus may improve the hardness of steel.

The diagram of partitioning time versus volume fraction of retained austenite and quenching temperature versus final carbon content in austenite is shown in Figure 4. In the present work, we did not take into account the volume fraction of carbides (Figure 4(a)). According to the diagram, it is indicated that with the increasing partitioning time, the volume fraction of the retained austenite first increased and then decreased, similarly the final carbon content in austenite first increased and then decreased. According to the CCE model [11] proposed by Speer and the Magee–Koistinen–Marburger formula [12],

$$f_a = 1 - \exp\left[-1.10 \times 10^{-2} \times (M_s - T_q)\right].$$

In the above formula, $f_a$, $M_s$, and $T_q$ is the volume fraction of retained austenite, the start temperature of martensite, and the quenching temperature, respectively.

When the quenching temperature was 210°C, the volume fraction of retained austenite was 43%. However, there are some differences between theoretical calculations and the results obtained by the means of XRD. The experimental measurement was much lower than the theoretical calculation, and at the same partitioning temperature, the content of retained austenite was different for different partitioning times. In the present work, the carbon partitioning process was divided into two stages. With the partitioning time increasing from 10 to 600 s, the content of retained austenite increased gradually, and it reached the maximum value at 600 s. Subsequently, with the increasing partitioning time, the content of residual austenite decreased. In the first stage, the partitioning time was not enough for the partitioning of carbon, therefore the retained austenite was not stable, and the content was low. In the second stage, the amount of carbide precipitates increased with increasing partitioning time, therefore the carbon content of austenite decreased, that made the content of retained austenite lower at room temperature.

The plot of partitioning-tempering time against hardness for alloy 1 and alloy 2 is shown in Figure 5(a). The hardness of alloy 1 and alloy 2 first decreased, then increased at 1800 s, and then decreased again. It is interesting to note that there was a peak value of hardness for the Nb-containing steel (about 450 HV1) and Nb + Cu-containing steel (about 461 HV1) at the partitioning-tempering time of 1800 s. It is seen that the hardness of Nb-containing steel was lower than that of Nb + Cu-containing steel. At a certain partitioning-tempering time, the peak value of hardness means that the precipitation is critical. When the partitioning-tempering time is more than the peak value time, the increasing amount of precipitates is compensated for by the
growth of the precipitated particles, therefore the precipitation hardness is decreased with increasing particle size. The calculated equilibrium phase fraction of carbides for the investigated steels, as calculated by JMatPro, is shown in Figure 5(b). It can be seen that fine carbides such as M(C, N), M7C3 and Cu are precipitated within a wide range of temperature. Nb can form stable compounds with C and N. Very fine ε-Cu is known to be precipitated and contributes to the precipitation hardening of steels. The reason is that the copper particle is softer than the steel matrix, which may strengthen the matrix without decreasing the plasticity significantly.

Conclusions
The designed medium carbon steels treated by Q-P-T process was investigated and the conclusions can be drawn as follows:

(1) the microstructure of the Q-P-T processed medium carbon steels mainly composed of nanoscale martensite and retained austenite;
(2) owing to the addition of copper, the hardness, volume fraction of retained austenite and final carbon content in the retained austenite for the Nb + Cu-containing steel were higher than the Nb-containing steel. This was because the precipitation of Nb was promoted by the addition of Cu.

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No potential conflict of interest was reported by the authors.

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