Characteristics of Retained Austenite in Quenched High C-High Cr Alloy Steels

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In this paper, the morphologies of martensite and retained austenite for 1.5 mass%C-12 mass%Cr and 1 mass%C-8 mass%Cr steels were observed by means of optical microscopy, XRD, SEM/EBSD and TEM. The amount of retained austenites was quantitatively investigated and compared with XRD, EBSD and TEM observation methods. The retained austenites were distributed in the form of a block type and a film type in the martensite structures. For the 1.5 mass%C-12 mass%Cr steel, the amount of block type retained austenite and film type one are almost equal in three distinct regions of carbide; primary carbide, secondary carbide and without carbide. In the case of 1 mass%C-8 mass%Cr steel, as to film type retained austenite there are not so much differences among those regions. However, block type retained austenites are distributed with much amount, especially in the region around primary carbide. [doi:10.2320/matertrans.MRA2008161]

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1. Introduction

High C-high Cr alloy steels are widely used around the world as a material for a great many dies and tools in cold press forming and cold forging of parts for automobiles, electronics and electrical appliances. In the case of cold working process, these tools, punches and dies subject to repeated usage require material quality featuring wear resistance, compressive strength, toughness and fatigue strength.

The microstructure prior to quenching consists of ferrite matrix and large-grained primary carbide M₁₂C₃, smaller secondary carbide M₂₃C₆ in these steels. The total amount of those primary and secondary carbides is 10–20% of the volume fraction in this type material and the actual amount of carbides depends upon casting conditions, hot working, heat treatment conditions, etc. as related to the manufacturing process.

These alloy steels are quenched and tempered before usage and their material properties are greatly influenced by the characteristics of the alloy carbides. Accordingly, the researchers to date¹⁻⁶ have generally considered the uniform dispersion and fineness of M₁₂C₃ and M₂₃C₆. Meanwhile, very few studies⁷⁻¹⁰ have been carried out on the properties and characteristics of the matrix structure itself, which comprises 80–90% of the volume ratio of the microstructure. The another reason why the studies of matrix structures in high C-high Cr alloy steels have rarely been performed is that the changes of structures in their transformation are very complicated and such kind of studies on “matrix” have not been needed up to now. From now on, the necessity for new studies on matrix structure in high C-high Cr alloy tool steel will be greatly increased in proportion to the needs of better performance in cold work tools and dies.

Two kinds of steels, 1.5 mass%C-12 mass%Cr and 1 mass%C-8 mass%Cr alloy tool steels are well-known. We abbreviate these alloys as 12Cr steel and 8Cr steel hereafter. The former has been, for long time, the representative steel in cold work tool steel group and the latter has lately come to be used in a domestic market and an overseas market in this cold work field.

In previous papers,¹⁰,¹¹ we reported the difference in the morphology of martensite and retained austenite depending on austenitizing temperature and solute carbon in matrix. The film type retained austenites were found at the lath boundaries for 12Cr and 8Cr steel, and block type retained austenites were found for 8Cr steel. In order to understand characteristics of retained austenite, it is necessary to understand the distribution of retained austenite around carbide. In this paper, precise distribution of retained austenites was studied in the regions around primary carbide, secondary carbide and without carbide for the 12Cr and 8Cr steels that were quenched.

2. Experimental Procedure

The chemical compositions of the specimens are indicated in Table 1. Following hot forging, the billet was hot-rolled into round bars, 80 mm in diameter and was then performed to spheroidization. The specimen cubes measuring 10 mm on each side were cut from round bar cross sections at the middle of the bar and the circumference, in the rolling direction. Each specimen was maintained for 10 min at an austenitizing temperature, ranging from 950°C to 1200°C, in 50°C intervals, and was quenched by subsequent air cooling.

| Table 1 Chemical compositions of the steels used (mass%). |
|------------------|------|------|------|------|------|------|------|
|                  | C    | Si   | Mn   | Cr   | Mo   | V    | Fe   |
| 12Cr steel       | 1.40 | 0.23 | 0.42 | 11.59| 0.80 | 0.23 | Bal. |
| 8Cr steel        | 0.96 | 0.98 | 0.41 | 7.23 | 1.93 | 0.24 | Bal. |

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It should be noted that the temperature, 950°C is approximately 150°C higher than the $A_1$ transformation temperature and that 1200°C is in the vicinity of the eutectic temperature in these steels.

The amount of retained austenite and the lattice parameter were measured by means of XRD for the quenched specimens. The measurement condition with regard to XRD is that the scanning speed is 2°/min and measured planes are (200)\textsubscript{\beta}, (220)\textsubscript{\gamma}, (211)\textsubscript{\beta} and (311)\textsubscript{\gamma}. The amount of retained austenite is estimated using intensity ratio of $\alpha$(martensite) and $\gamma$(austenite). EBSD is useful for observing crystal orientation. Thus SEM/EBSD was used to observe the morphology of packet of martensite and retained austenite. The morphologies of martensite and retained austenite were observed by optical microscope and TEM. From the dark field images of TEM, retained austenites were observed and the volume fractions of retained austenite were measured.

3. Results and Discussion

3.1 Structural observation by optical microscope

Figure 1 shows the optical microstructures quenched from austenitizing temperature 1050°C and 1150°C in 12Cr steel. As the austenitizing temperature rises from 1050 to 1150°C, the fine, secondary carbides proceed to dissolve into matrix. The pinning effect which limits grain boundary movement is reduced. Thus the coarsening of the austenite grains can be found. The typical value of austenite grain size for 950°C austenitizing temperature is 4–5 μm for both steels. These values increased rapidly above 1100°C austenitizing temperature, 20 μm in 12Cr steel and 90 μm in 8Cr steel austenitizing at 1200°C. The martensite quenched from 1150°C has lenticular type of martensite, and that from 1050°C has a martensite laths which has fine structure compared with the lenticular type martensite. The change in morphology of martensite for high C-high Cr steels is similar to carbon steel.\textsuperscript{12)}

3.2 Amount of retained austenite measured by XRD

The amounts of retained austenite measured by XRD for 12Cr and 8Cr steels quenched at each austenitizing temperature are indicated in Fig. 2.\textsuperscript{10} The volume of retained austenite is 5% at 950°C and increases to 30% at 1100°C, and then reaches approximately 60% at 1200°C. Therefore it can be understood that retained austenite at room temperature becomes more stable with increasing austenitizing temperature.

As the austenitizing temperature increases, the solution of carbides into the austenite matrix proceeds and the carbon concentration in the matrix increases. Computation of the carbon concentration in its matrix using Thermocalc\textsuperscript{10} results in 0.44 mass% for 12Cr and 0.45 mass% for 8Cr at 950°C, increasing to 0.90 mass% and 0.91 mass% respectively at 1150°C. Accordingly, it can be seen how influenced the amounts of carbon solution into matrix to greater chemical stability of austenite.

3.3 Change in the austenite lattice parameter

Figure 3 shows the lattice parameters of austenite in 12Cr steel and 8Cr steel with XRD measurements. It can be understood these lattice parameters are influenced by austenitizing temperature. The lattice parameter is 0.3590 nm at 950°C, rising up to 0.3605 nm when the austenitizing temperature reaches 1200°C. The average
amounts of carbon concentration in austenite matrix for both steels at 950°C and 1200°C are 0.45 and 1.00 mass% respectively according to Thermocalc. These lattice parameters appear somewhat large in comparison with the 0.3595 nm for 1.0 mass% carbon steel as reported by Honda and Nishiyama. This may be ascribed to the addition effect of Mo, which atomic radius of Mo is larger than that of Fe. Not only the effect of Mo, but also carbon atoms must give an effect of increasing lattice parameters. With increase of austenitizing temperature, secondary carbide $M_23C_6$ proceeds to dissolve into austenite matrix. As a result of more solution of carbon atoms, they become to enter into the specified positions by interstice, therefore the austenite lattice parameter is expanded.

### 3.4 Structural observation and amount of retained austenite measured by EBSD

In comparison with carbon steel and low alloy steel, high C-high Cr alloy steels do not have completely uniform structures because of their complicated dispersion of primary carbides and secondary carbides. Three regions, which are distinguished from carbide, are considered; that is, (i) around primary carbide, (ii) secondary carbide and (iii) without carbide regions. EBSD observations were carried out around these regions. The observed specimens were selected just for lath martensite structure quenched from 1050°C because of focusing on the practical use.

The EBSD results are shown in Fig. 4, which indicated packet map in martensite structure, on upper side and dispersion of retained austenite ($\gamma_R$) map, on lower side. Since the dispersion of retained austenite for these three regions are similar each other for 12Cr steel, just secondary carbide regions are shown. The packet maps in martensite show that four kinds of packets are distributed, while retained austenite, whose sizes are 0.2–0.5 μm, are observed in block type at the packet boundaries, in comparison of upper and lower side.

The amount of retained austenite for 12Cr steel is 9.9–11.9 vol% and is almost equal in the three regions. Hence just one region around secondary carbide is shown here. The EBSD observations for 8Cr steel quenched at 1050°C showed similar structure of martensites to that of 12Cr, while the retained austenites are observed in the form of 0.1–0.5 μm blocks type at the packet boundaries. A major difference is found on the amount of retained austenite, that is, 19.4 vol% around primary carbide, 1.9 vol% around secondary carbide and 3.9 vol% without carbide region. Here, the amount of retained austenite measured with EBSD was assumed to be area percentage of retained austenite. Although they must have 3D structure, its ratio found in surface is considered to be not so far from volume fraction.

### 3.5 Structural observation and amount of retained austenite measured by TEM

TEM observations for the specimens of 12Cr and 8Cr
steels quenched at 1050°C were also carried out around the above three regions. The results are shown in Fig. 5. The morphologies of martensites structure for both steels were a lath type in bright field images.

The dark field images using (111) diffraction spot of austenite in 12Cr and 8Cr steel show the retained austenite is film type with width of 10–50 nm located at the martensite lath boundaries. The location of film type retained austenite is not related to the carbide dispersion.

The amount of retained austenite was measured as follows; a constant length of line perpendicular to the width of film type retained austenite was drawn and the fragment of width of the retained austenite occupied was regarded as volume fraction. Here, we assume the observation of retained austenite in 2D is the same as 3D. This is the same method to measure the width of lath in martensite structure shown by Rao.17) Width of retained austenite was measured with condition that the incident beam was parallel to the austenite boundary. The amount of austenite is about 10 several vol%.

The morphology of retained austenite was observed using EBSD and TEM as described above. EBSD observation is suitable for lager retained austenite, like block type, and TEM one is necessary for rather smaller retained austenite, like film type. Therefore, if the amount of retained austenite is measured by EBSD without TEM, that of value will be lower than the proper one.

![Fig. 5 TEM bright field images for 12Cr steel and 8Cr steel austenitized at 1050°C are shown in upper part of figure. Dark-field images are shown in lower part. The film type austenites were found at lath boundaries. Volume% of retained austenite ($\gamma_R$) are shown in figures.](image)

![Fig. 6 Amount of retained austenite measured with EBSD+TEM method in the regions surrounding primary carbide (i), secondary carbide (ii) and in the region with no carbide (iii). (iv) indicates the amount of retained austenite measured with XRD method.](image)
retained austenite value from XRD corresponds well with the sum of EBSD results in hatched and TEM ones in black. In the case of 8Cr steel, however, the amount of retained austenite in the vicinity of primary carbide is much more, while the amounts of retained austenite in the other two regions are lower. The total amounts of retained austenite vary depending on carbide regions. It is necessary to consider and determine the distribution of three regions in order to compare XRD value. These phenomena between 12Cr steel and 8Cr steel may be considered to be due to the differences in the amount of secondary carbide and differences in Si content which delays the diffusion rate of C.

4. Conclusions

The morphologies of retained austenite were observed with TEM and SEM/EBSD in the three distinct regions around primary carbide, secondary carbide and without carbide regions for the 12Cr and 8Cr tool steels that were quenched. The amount of retained austenite was measured with TEM, SEM/EBSD and XRD and was compared each other. XRD was utilized for determining lattice parameters at various austenitizing temperatures.

(1) Accompanying increased austenitizing temperature, carbon in alloy carbides dissolves into austenite matrix and the amount of retained austenite rises to a maximum of about 60 vol% at 1200°C. The austenite lattice parameter associated with the amount of carbon solution in matrix increases from 0.3590 nm to 0.3605 nm.

(2) Both block and film types morphologies of the retained austenite are distributed simultaneously in 12Cr steel and 8Cr steel quenched from 1050°C. As a result of EBSD investigations, block type retained austenite can be observed at the packet boundaries in martensite. TEM observations revealed that the film type retained austenite can be found at the lath boundaries of martensite.

(3) In the case of 12Cr steel, the amount of block type retained austenite and film type one is almost equal in three distinct regions. XRD value for the amount of retained austenite corresponds well with the sum of EBSD results and TEM ones.

In the case of 8Cr steel, as to film type retained austenite there are not so much differences among three distinct regions. However, block type retained austenite is distributed with much amount, in the region around primary carbide.

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