The Morphology and fine Structure of Martensite in Fe-Ni-C Alloys

H.L.Cao, Y.Y.Guo and X.M.Zhang, Institute of Metal Research, Academia Sinica, Shenyang, China

Introduction

The martensite transformation is a very widely studied subject in the region of material science. There have been plenty research works and theoritical evaluations being carried out as it is one of the basic phase transformations in the solide state metals. Since 1960s when P.K. Kelly and J. Nutting(1,2) first observed the substructure of martensite in thin foil, most investigations studying the martensite fine structure have been concentrated on employing the transmission electron microscope(TEM) while the use of optical microscopes is not that popular due to the limit of the optical resolution and the fine structure revealing method. In this decade the color metallographic technique has been rapidly devaloped so that provides a much wide application range of the optical microscopes. In this aspect we have carried out some experimental works(3,4). This study, on the bases of previous works, is to investigate the morphology, distribution and the fine structure of martensite as well as the colored grain orientation difference in three Fe-Ni-C alloys.

Experimental procedure

The alloys used in this work were 10kg ingods melted in a vacuum induction furnace and the chemical compositions are listed in table 1.

No.	Nominal comp.	Ni	C	Mn	Si	S	P	Fe
1	Fe-24Ni-0.2C	23.93	0.18	0.28	0.18	0.013	<0.05	balanced
2	Fe-24Ni-0.4C	24.05	0.36	0.24	0.19	0.011	<0.05	balanced
3	Fe-24Ni-0.8C	23.86	0.72	0.26	0.21	0.010	<0.05	balanced

Table 1: Chemical compositions of the alloys studied (wt%)

The ingods were hot-forged to 15mm thick plates, then relief-annealing at 1000°C for 1 hour followed by furnace cooling. The solution treatment was performed at 1100°C for 30 minutes, water quenched. Finally the specimens were held 20 minutes in liquid nitrogen(-196°C) for deep cooling treatment.

The metallographic specimens were prepared in traditional way (cutting to 15x15mm, grinding and polishing) and then etched by two-fold electrochemical etching procedure: a. 10% nitric acid in ethanol, 25°C, stainless steel(SS) cathode, 2-3v DC, 3 seconds; b. 15% sodium thiosulfade water solution, 25°C, SS cathode, 3-5v DC, 5 seconds. The first process is to outline the grain and/or phase boundaries while the following etching conducts a selective deposite film on the previously outlined surface, thus provides a distinctive color apperance of martensite observed by the optical microscope.

The etching pits were obtained by electrochemical etching performed under the

conditions of: 10% hydrochloric acid methanol solution, 20°C, SS cathode, 1.5v DC, 5.5 minutes. The scanning electron microscope(SEM) was employed to analyse the etching pits geometry.

Experimental results and discussions

As well known, the morphology of martensite is strongly depended on the Ms temperature while the Ms temperature is mainly effected by the chemical composition of the alloy, especially the carbon content. In this study three Fe-Ni alloys containing different carbon contents were used and the morphologies changed with the increase of carbon contents. Figure 1 illustrates the microstructure of alloy 1(0.2C, wt%). Obviously the whole image appeares to be the packed-lath-martensite morphology. In fact, the Ms temperature of this alloy is above the room temperature and these packed-lath-martensites have formed by water quenching. The alloy 2 shows a complex morphology consisting of two types of but three morphologically different martensites(Fig. 2): type 1 -- the lath-martensite block-distributing in the matrix and type 2 -- a. high temperature butterfly-martensite(bright-grey in Fig. 2) which formed during the water quenching, i.e. Ms temperature being above the room temperature, this has also been varified by a parallel study recently(5); -- b. low temperature butterfly- martensite(dark-redish in Fig. 2) which transformed during the deep cooling treatment in liquid nitrogen. The two-fold electrochemical etching process is quite an effective metallographic revealing technique which has conducted a distinguished morphological appearance of the mixed martensite microstructures.

Fig. 1: The packed-lath-martensite microstructure of alloy 1 (0.2C, wt%)

Fig. 2: Mixed morphologies of alloy 2(0.4C, wt%), lath-martensite(matrix), high temperature butterfly-martensite (a) and low temperature butterfly-martensite(b)

Fig. 3: The grain orientation difference in coloring and cross-boundary martensite in alloy 3(0.8C, wt%)

In the study of alloy 3(0.8C, wt%), three interesting phenomena were noticed. First, on the seccess of revealing austenite grains in different colors, the cross-boundary martensite was found(Fig. 3). This is a very surprising phenomenen which can hardly be explained by the traditional martensitic transformation model. No evidance has been found by TEM examination, probably due to the large grain size of the specimen and/or the lack of observed fields in TEM examination. Further TEM work is going on and hopefully could carry out some significant results to explain the mechanism of the cross-boundary martensite formation. Second, the bended-twins substructure existed in the large lenticular martensite(Fig. 4). The other research work(6) has indicated that in Fe-Ni-C alloy the large platelike martensite with $(252)_f$ habit plane consists of lots of small platelets, i.e. the small platelets with the same orientation could join together along the $(011)//(111)_f$ plane to form the large martensite plate. This is in line with the microstructure observed in alloy 3 of this work. Since the formation-rate of lenticular martensite is very high(about 1-2km/s), a formed martensite platelet could produce a compressive stress on the untransformed matrix in front. If there are some deffects or alloy segregations existed in this area, an uneven growth of the next platelet could occur, thus continuing to produce a gradually bended-twins substructure of the large lenticular martensite. Furthermore, three morphologically different martensites can be observed in alloy 3(Fig, 4): a. bended-twins lenticular martensite, b. small twinning lenticular martensite and c. thin plate(niddle) martensite. The former should be the one occuring first at Ms temperature and the middle type forms during the deep cooling while the later is the product at very low temperature. M. Maki et al.(7) have pointed out that the morphology of martensites in Fe-Ni-C alloy varied at -150°C from lenticular to thin plate with no midribs.

Fig. 4: Bended-twins substructure of large lenticular martensite(a), small twinning lenticular martensite(b) and thin plate martensite(c) in alloy 3(0.8C, wt%)

The color difference from grain to grain as shown in Fig. 3 has been considered as the orientation difference and this has been evaluated by SEM analysis of the geometry of etching pits. The symetric square pits obtained in alloy 3 (0.8C, wt%) were imaged by turning its diagonal paralell to the tilting axis of the specimen holder(Fig. 5a) and then the tilting operation was continuously performed from zero position until one of the adjacent walls was vertical(Fig. 5b). The geometry of etching pits is schematically illustrated in Fig. 5c. The experimentally tilted angle was just 45 degrees and the angle between the vertical adjacent walls, θ, was measured to be 120 degrees. The angle between the opposite walls, ϕ, can be calculated by the following equation

$$\cos\phi = 1 + 2\cos\theta$$

where $\theta = 120$, so that $\phi = 90$ is obtained. In FCC austenite the angle between (110) planes are 60, 90, 120 or 180 degrees. Thus the pits walls in Fig. 5 are consisted of (110) planes and no other orientation could satisfy this condition, i.e. the etching pits evaluated represent (110) plane and the orientation of the austenite grain is (100)γ. By such evaluation combined to electrochemical etching process, the corelation between the grain orientation and the etching colors could be estimated so that provides a practical way to study the martensitic transformation metallographically by the optical microscopy.

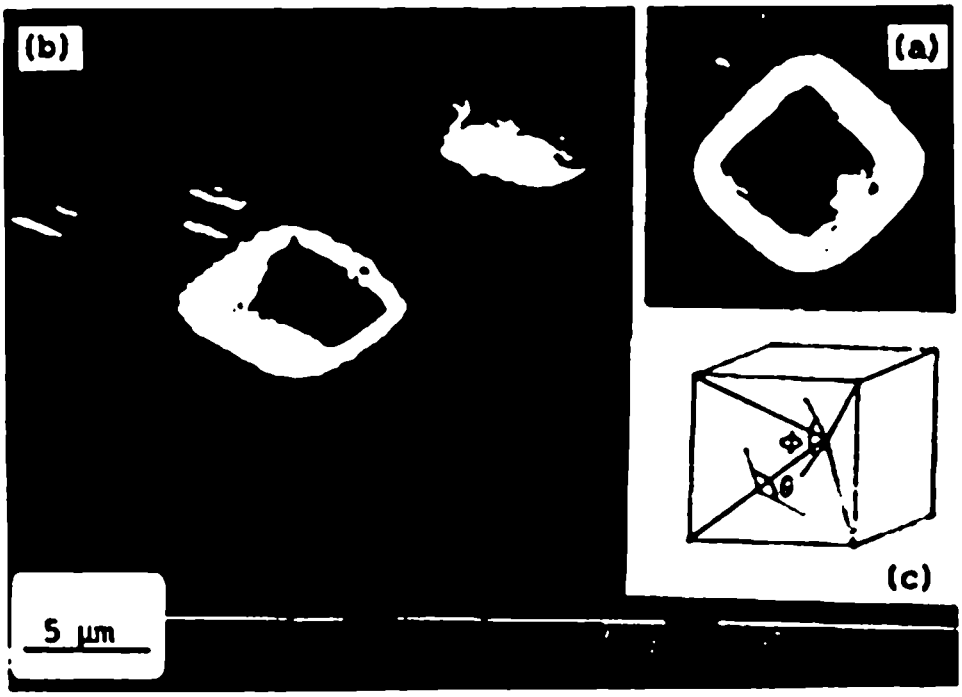

Fig. 5: Etching pits geometry analysis of alloy 3 (0.8C, wt%) by SEM. a. second electron image, zero position; b. the image after tilting 45 degrees from zero position; c. schematic illustration of etching pits geometry

Conclusions

1. Four types of martensites have been obtained by varying the carbon contents from 0.2 to 0.8(wt%) in Fe-24Ni alloys. The morphology of martensites changes with increasing carbon contents in the order of lath -- lath plus butterfly -- lenticular plus thin plate(niddle).
2. By two-fold electrochemical etching, both the lenticular martensite substructure(twins) and the grain orientation difference have been revealed in Fe-24Ni-0.8C alloy, sequentially the cross-boundary martensite has been found.
3. Bended-twins substructure was observed on the lenticular martensite as a result of uneven stimulative growth of martensite platelets in the area where deffects and alloy segregations existed.
4. The eveluation of the etching pits geometry conbined to electrochemical color etching can be a comprehensive method to estimate the corelation between the grain orientation and etching colors thus to provide a practical way for studying the martensitic transformation metallographically by the optical microcope.

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