

Identification of Bainite in a Multi-phase Microstructure of an Austempered Steel Alloy: A Metallography Approach

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ABSTRACT – Structural characterization of a multi-phase steel has become an exciting issue in various studies to date. This relates to the difficulty in distinguishing phases with similar morphology, i.e. bainite and martensite, through chemical etching. This study discusses a method to observe bainite phase through a metallographic approach on FeNi steel using color etching. Variations in the use of etching in this research include 2% nital, 4% picral, and 15% sodium metabisulphite (SMB). First, the samples were austenized then austempered at either 400 °C or 500 °C, for 60 min followed by quenching in either water or brine solution. Based on optical microscope observations, SMB color etching provides more explicit information on the visualization of bainite and martensite phases because they have different color appearances. The bainite phase is shown in bluish color, while the martensite phase is shown in brownish color. Furthermore, the influence of variation in austempering temperature and quench media on microstructure morphology was also discussed. In addition, the calculation of the lattice parameters of the X-Ray Diffraction (XRD) pattern was also carried out in this study to identify the crystal structure formed.

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INTRODUCTION

Bainite is a phase in the steel formed from the austenite phase transformation process through a heat treatment mechanism known as the austemper process [1–4]. The transformation process occurs above the initial temperature of the formation of martensite (martensite start) and below the formation of a delicate pearlite phase. The presence of bainite in steel alloys provides an advantage in increasing the strength of the alloy without requiring high production costs [5]. So that in its application, steel alloys with the bainite phase are often used in the rail transportation industry [6–7] and automotive [8]. Depending on the process flow used, bainite can be found as the dominant phase [9–10], or as one part of a multi-phase system [11–13]. In alloys with multi-phase systems, not only bainite is formed, but it is also possible to form other phases such as martensite, residual austenite, and ferrite.

One of the issues that are still an interesting discussion in observing the formation of the bainite phase in steel alloys is the initial identification of the phases in the final microstructure formed. With the various process flows to obtain the bainite phase, the resulting bainite phase can have different morphologies and distributions. Furthermore, visual observation of this microstructure can carry out using several methods, among others, from optical microscopy to electron microscopy [5, 14–16].

Although the metallographic method with an optical microscope is a relatively inexpensive method with relatively easy sample preparation to identify the phase properly, selecting an appropriate solution and etching process is necessary. The most commonly used etching solution for steel alloys is nitric acid with ethanol (nital) with a specific concentration that varies according to needs [17]. Nital works by attacking the grain boundaries of ferrite and martensite phases [18]. Unfortunately, the process of characterizing multi-phase steel alloys containing bainite and martensite cannot be accommodated if it only relies on a nital etching solution.

Therefore, in this study, observations of bainite in FeNi multi-phase steel alloys through metallographic methods were carried out through the use of several etching processes, both non-colored and colored. In addition, hardness testing and analysis of X-Ray diffraction patterns were conducted to support data in identifying the phases formed in FeNi multi-phase steel alloys.

EXPERIMENTAL METHOD

Materials and Instruments

The sample used in this study is an as-cast FeNi low carbon steel with chemical composition shown in Table 1. This chemical composition was obtained from Optical Emission Spectroscopy (OES) Bruker Q4 Tasman.

Table 1. Chemical composition of FeNi Steel (% wt)

C	Si	Al	Mn	Cr	Mo	Ni	P	S	Fe
0,28	1,41	0,98	0,4	0,09	0,17	4,49	0,0005	0,0003	Bal.

Method and Procedure

The sample was subjected to a series of heat-treatment process to obtain a bainite contained multi-phase structure. The experimental method of this study is shown in Figure 1. The heat treatment process consists of an austenizing process at 950 °C for 60 minutes, followed by an austempering process at either, 400 °C or 500 °C, for 60 minutes using a Nabertherm C-440 muffle furnace. Afterward, the quenching process is performed using two cooling medias: water and brine solution cooling media to get the estimated 45-90 °C/sec of cooling rate target. The heat treatment process temperature is determined using the JMat Pro Version 11 software simulation.

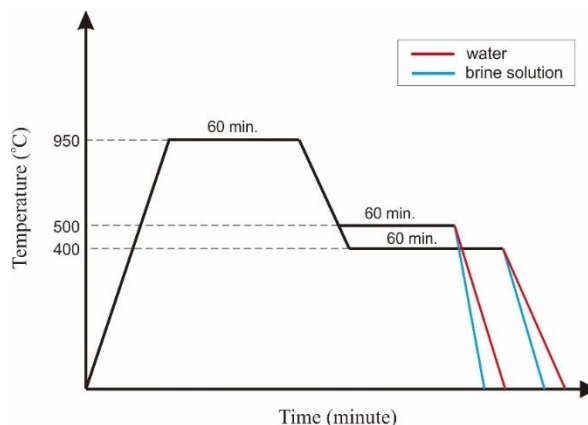
From the previously mentioned experimental method, there are four types of samples observed. These types are distinguished based on the austempering temperature and the cooling media. Table 2 shows the code for each sample in this study.

The heat-treated samples were then prepared for hardness testing using the AFFRI 206 RTD Rockwell scale machine and followed by the metallographic analysis process using an Olympus BX-53M optical microscope.

Metallographic preparation begins with the process of cutting the sample to the desired size. The cut samples are then mounted using resin. The following process is grinding with sequential roughness starting from 80#, 120#, 200#, 400#, 800#, and 1000#. The next step is polishing with a roughness step of 3 and 1 micron to produce a mirror surface. The last step is etching with several combinations of methods, including rubbing and immersion.

Metallographic analysis was carried out using several etching solutions to identify the formed phase, especially the bainite phase. After that, the visual approach was performed to compare each solution's etching results. The etching solution used in this study is presented in Table 3.

The formed crystal structure analysis by X-Ray Diffraction (XRD) was also performed using a Bruker D8 Advance 3kW engine with a Lynx Eye XE-T detector and a Cu K- α radiation source (wavelength: 1.5406). The range of measurement angles carried out is 0-120°.

**Figure 1.** Heat treatment process diagram**Table 2.** Identity of samples

Code	Treatment
A4	Water quenching, austempering temperature at 400 °C
G4	Brine quenching, austempering temperature at 400 °C
A5	Water quenching, austempering temperature at 500 °C
G5	Brine quenching, austempering temperature at 500 °C

Table 3. Variation of etching solution

Etch Solution	Chemical composition
Nital 2%	HNO ₃ (2 ml) + ethanol (100 ml)
Picral 4%	Picric Acid (4 gr) + ethanol (100 ml)
SMB 15%	Sodium metabisulphite (15 gr) + aquades (100 ml)

RESULT AND DISCUSSION

Microstructure Analysis

Application of etching solutions is commonly in metallographic analysis to reveal microstructural patterns through chemical reaction processes. Several etching solutions were nital 2%, picral 4%, and SMB 15%. Each etching solution provides information on the different microstructure patterns of the formed phase. Figure 2 shows the microstructure of all as-quenched samples after etched with three different etchants. Samples etched with 2% nital (Fig. 2a, 2d, 2g and 2j) and 4% picral (Fig. 2b, 2e, 2h and 2k) reveal a slight similar microstructural features of the samples. Between these two etchants, the 2% nital samples show a more explicit grain boundaries than the microstructure in 4% picral samples. This is due to their preferential difference on the corroded area during sample surface microstructural revelation. Nital is intended to attack the grain boundaries of the ferrite microstructure on iron or steel materials, while picral has the primary function to attack the carbide phases, causing the visual appearance of the carbide to look more contrasting [19]. For the FeNi samples, even though nital and picral can reveal the presence of ferrite grains (Fig. 2a, 2d, 2g and 2j) and lath-like carbides (Fig. 2b, 2e, 2h and 2k) throughout the sample, respectively, it is still challenging to determine more specifically the phases in each sample from these two etchants.

The use of color etching in multi-phase alloys can facilitate the identification of phases in an alloy [19–22]. Color etching becomes a choice because it can display different visualizations through contrasting colors in each phase formed. It also can mark off bainite and martensite phases more clearly, which are generally tricky to distinguish using ordinary etching because of similar microstructure characteristics.

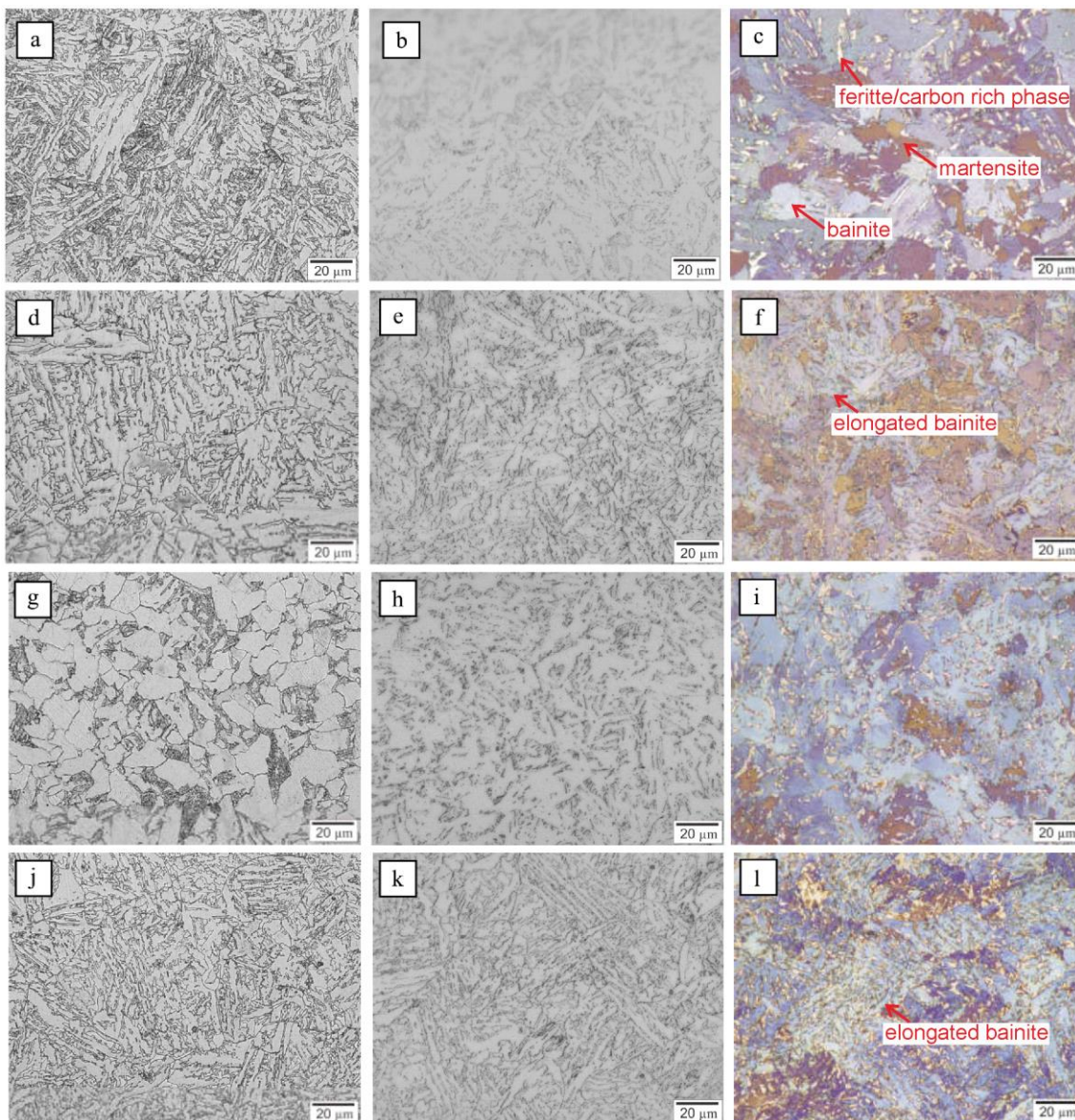


Figure 2. Microstructure of FeNi: (a) A4 nital etch 2%; (b) A4 picral etch 4%; (c) A4 SMB etch 15%; (d) A5 nital etch 2%; (e) A5 picral etch 4%; (f) A5 SMB etch 15%; (g) G4 nital etch 2%; (h) G4 picral etch 4%; (i) G4 SMB etch 15%; (j) G5 nital etch 2%; (k) G5 picral etch 4%; and (l) G5 SMB etch 15%

Table 4. Area fraction of phases

Sample Code	Area Fraction (%)		
	Ferrite (white area)	Bainite (blue area)	Martensite (brown area)
A4	31.34	48.11	20.55
G4	12.12	67.34	20.54
A5	24.52	51.84	23.64
G5	6.11	70.48	23.41

The microstructure images of all samples using SMB etchant are shown in Figure 2c, 2f, 2i and 2l. It shows a complete color visualization of the microstructure instead of black and white as in the nital samples and picral samples. Each color resulted from etching with SMB represent a different phase. The brown, blue, and white colors on the SMB etching results represent the martensite, bainite, ferrite, or residual austenite phases, respectively [23-24]. A similar statement was also conveyed by Medonca et al. that the white area shown in the SMB etching results traces the austenite phase or inclusions that were not detected [25]. While the presence of a yellowish area on the microstructure indicates a fairly high carbon content, this corresponds with the research conducted by Krugla et al. [26]. Each phase formed through SMB etching can be identified more quickly through the color differences shown. Other information obtained from SMB etching is that both brine solution and water-cooled samples show ferrite, bainite, and martensite phases. Visually, the presence of the bainite phase is more dominant than the other phases by showing the sizeable blue color area. Further analysis using ImageJ software shows the area fraction of phases depicted on table 4.

Color etching has a different way of working with corrosive types of etching (nital and picral). Color etching will result in film deposition on the sample surface, so it needs careful handling so that the film layer is not damaged when observed. Over time, the film layer will change color so that, in this case, it causes color differences in the observed samples, including bright blue, dark blue, or purplish-blue. Further explanation of the phase formed using XRD analysis will be presented in the subsequent discussion.

Furthermore, the austempering temperature shows its effect on the grain characteristics of the sample. Qualitatively, austempering at 500 °C introduces a finer grain size (in both the white and blue areas) than samples with heating at 400°C. The bainite phase also tends to form elongated with contrasting boundaries as the cooling rate increases. This condition may occur due to the difference in austempering temperature, which affects the phase transformation mechanism during the isothermal process. Previous studies have asserted that the austempering temperature will impact the size and shape of the grains [27].

XRD Analysis

XRD analysis provides information regarding the crystal structure of a phase. Based on the results of the XRD analysis in Figure 3, the diffraction peaks of the sample consist of {110}; {200}; {211}; {220}; {310}. The crystal structure formed in most diffraction peaks is a cubic structure (BCC-FCC), and there is also a tetragonal structure (BCT). The formation of a tetragonal structure in steel indicates the existence of a bainite or martensite phase. The cubic and tetragonal structure can be identified by performing manual calculations using the Bragg equation to show the value of the lattice parameters a, b, and c. In general, the tetragonal structure has characteristic lattice parameter values $a = b \neq c$. Table 5 shows the lattice parameter values for each sample. In this case, lattice parameter information clarifies the ambiguity of the bainite phase identifying in the etching analysis in the previous chapter.

Furthermore, the diffraction peaks in the sample shown in Figure 3 do not indicate the formation of the residual austenite phase. In general, the existence of the residual austenite phase in steel samples is indicated by the gamma diffraction peak (γ) in the XRD analysis results based on the previous studies [4, 28].

Table 5. lattice parameter of FeNi samples

Sample Code	Precise Lattice Parameter (Å)		
	BCC	FCC	BCT
	a=b=c		a=b c
9A4	2.897	4.073	2.880 2.879
9G4	2.898	4.095	2.897 2.828
9A5	2.897	4.020	2.841 2.906
9G5	2.870	4.059	2.873 2.904

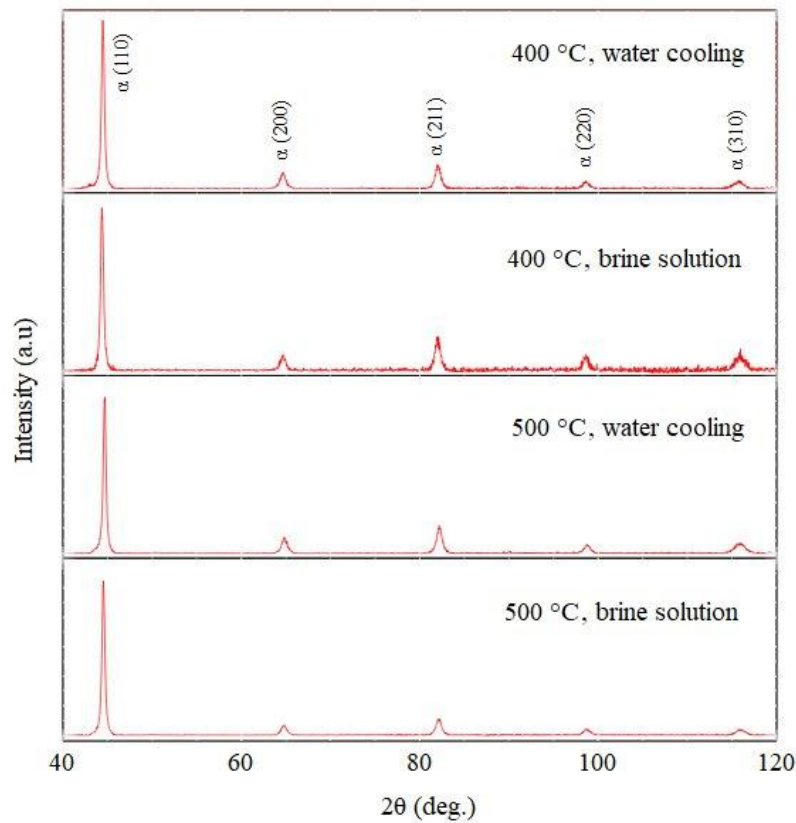


Figure 3. XRD analysis of FeNi samples

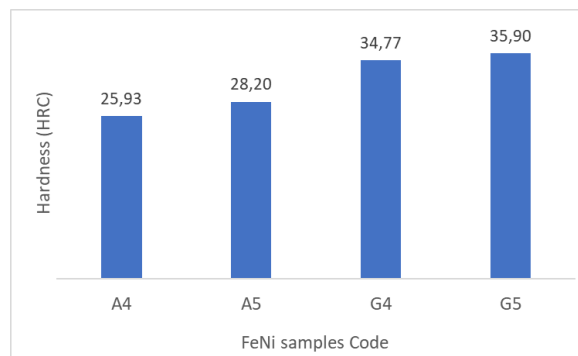


Figure 4. The hardness value of FeNi samples

Hardness Analysis

Hardness testing was carried out to determine the effect of austempering temperature and cooling media on the hardness value of FeNi samples. The results show an increase in the hardness value due to an increase in the austempering temperature and cooling rate, as shown in Figure 4. This phenomenon corresponds due to the formation of bainite and martensite phase as the austempering product.

Previous research stated that variations in austempering temperature would produce different phases and influence the mechanical properties. Austempering at a temperature of more than 400 °C tends to form bainite and tempered martensite. Furthermore, the tempered martensite phase contains much higher ϵ -carbide precipitates than the other phases. The presence of ϵ -carbide precipitates exhibits hardness improvement through dispersion strengthening [29]. Even though the samples have almost the same martensite area fraction, the difference of hardness distribution will still exist due to the influence of other dominant phase area fractions.

CONCLUSION

Phase identification in the studied samples is easier to do by using SMB 15% color etching. Each color represents the phase formed, including brown for the martensite phase and blue for the bainite phase. Although some studies have stated that the area represents the ferrite and carbon-rich phases, the whitish area is quite interesting to note. The sample with a austempering temperature of 500 °C has a more delicate grain morphology than the sample with a austempering

temperature of 400 °C. This phenomenon occurred because the phase transformation mechanism involves grain growth and is strongly influenced by temperature during the isothermal process. The XRD data represent that the crystal structure formed in the FeNi sample consists of cubic (BCC/FCC) and tetragonal (BCT) structures. The presence of a tetragonal structure indicates the existence of a martensite or bainite phase. Furthermore, the hardness distribution shows that the hardness of the FeNi steel sample is influenced by the area fraction distribution of phases after austempering process.

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