Technical Note



# A simple metallographic technique for revealing prior-austenite grain boundaries in CK45 steel

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## Abstract

CK45 steel is medium carbon steel that has a wide range of applications in the production of widely used parts such as shafts, gears, axles, crankshafts, forging, rails, etc. Austenitizing is the first step in most heat treatment processes for steel. Revealing austenite grain boundaries is very important because the austenite grains have a significant influence on steel properties. Because no research has been done on the detection of prior-austenite grain boundaries of CK45 steel, our main aim in this paper is to reveal the prior-austenite grains of this steel. We investigated two methods of chemical etching and oxidation etching to reveal prior-austenite grains boundaries of CK45 steel. Samples of CK45 with different structures(martensite, bainite, ferrite-perlite) steel were examined, and the optical microscopic images showed that the boundaries of the austenite grains were well visible with oxidation etching while with the chemical etching method, only the structure of the steels was determined.

*Keywords:* CK45 steel, Prior austenite grain boundary, Oxidation etching, Microstructure.

## 1. Introduction

Medium carbon steels are carbon steels that usually have a carbon content of between 0.30-0.55 % and a manganese content of between 0.60-1.65 % [1]. The most common use of medium carbon steels is in the manufacture of shafts, gears, axles, crankshafts, forging, rail axles, and railway wheels [2]. CK45 carbon steel is medium carbon steel that is widely used in various industries such as auto and engine parts and due to its low cost, this steel is regularly used in the manufacture of pump shafts and hydraulic clamps [3, 4]. For metallurgists to make high-quality steels, appropriate metallographic techniques are needed to characterize microstructural features such as phases, grain boundaries, and precipitates [5]. Austenitizing is the first step in most heat treatment processes of steel [6] also determining the prior-austenite grain size (PAGS) in steel is very important because of its effect on the transformation taking place during cooling, and thus, on the final properties. In fact, because of its significant impact on microstructure and mechanical, electrical, and magnetical properties, the accurate determination of the prior-austenite grain size of steels has received a lot of attention in metallurgical studies. Since new steels come to market every year, dependable methods for revealing the austenite grain boundaries in a wide range of steel compositions are needed [7–10]. Many methods have been used for revealing the austenite grain boundaries in steels [11]. In general, the methods of revealing austenite grain boundaries are divided into two categories: a) Direct methods: Use a high-temperature microscope to observe austenite grain boundaries at temperatures above the critical point, $A_{c3}$; b) Indirect methods: Methods for observing austenite boundaries at room temperature, the most important of these methods are the following: Chemical Etching (CE) method [12,13], Carburization method [14-16], Oxidation etching method [17], Thermal etching (TE) method [18]. Among various methods to reveal prior-austenite grains, chemical etch-

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ing is one of the most practical and widely performed methods [19], but revealing austenite grain boundaries by chemical etching could be a difficult task, especially for medium-carbon steel that showed sensibility to chemical etching [20]. In this work, we investigated two methods for the detection of prior-austenite grains in medium-carbon steel. The first method is to use an etchant based on picric acid, which has been used to reveal the primary austenite grains of several types of steels. In the second method, we used oxidation etching to reveal prior-austenite grain boundaries. The oxidation etching method involves heating a polished surface of the sample at austenitization temperatures in a furnace with an oxidizing atmosphere where the grain boundaries are revealed either by oxide accumulation or by carburizing the grain boundaries. Each of these methods was used separately to reveal prior-austenite grain boundaries in three medium-carbon steel sheets with different structures.

## 2. Materials and experimental procedure

The chemical composition of the steel is given in Table 1. To reveal the austenite grain boundaries, the specimens were first cut by a cutter (BUEHLER, America) of a CK45 bar. Six machined cylindrical specimens of 10 mm in length and 12 mm in diameter were used for this examination.

### 2.1. The first method: Chemical etching

In the first step, three of the samples were heat treated to obtain perlite, martensite, and bainite structures, then we used an etchant based on picric acid and a wetting agent for the detection of prior-austenite grain boundaries. The etching solution used consisted of 10 g of $CrO_3$, 50 g of NaOH, 1.5 g of picric acid, and 100 ml of distilled water. To make this solution, we first added $CrO_3$ to distilled water and then added sodium hydroxide(NaOH). The addition of NaOH should be done slowly because it generates a lot of heat and finally we add picric acid to the solution. In this method, the samples do not need to be heated before etching. This solution should be used at 120°C temperature due to the release of toxic fumes under the hood.

### 2.2. The second method: Oxidation etching

First, the surfaces of the other three specimens were sanded and then polished vertically to the main axis of the cylinder using standard metallographic methods, so that the cross sections of the samples are well glossy. Polished specimens are heated at a rate of 14°C/min to the above austenitization temperature (815°C) in the furnace (TC-Nicr) with oxidizing atmosphere, and the specimens are held for 1h at this temperature. Then to obtain perlite, martensite, and bainite structures respectively, one of the specimens was exited of the furnace and cooled in air, the second specimen was cooled in oil and the third specimen was austempered. For austempering, the specimens cooled to 400°C and remained at this temperature for 2h(in molten salt). In the next step, etching to reveal austenite grain boundaries was carried out with Nital-2% solution (2 ml $HNO_3$ + 98 ml of ethanol ($C_2H_5OH$)) etchant. The metallographic survey of specimens was done with an optical microscope Nikon E 100.

## 3. Results and discussion

Figure 1(a, b, c) shows the images obtained for pearlite, martensite, and bainite structures for CK45 steel after using the chemical etching method. This method was suitable for detecting prior-austenite grains boundaries in a wide range of steels with different structures but was not suitable for the CK45 steel tested and nothing other than the structure of the samples was detected, as can be seen from Figure 1.

Table 1. Chemical compositions of CK45 carbon steel bars.

| Fe | C | Si | Mn | P | S |
|---|---|---|---|---|---|
| balance | (0.42–0.5)% | 0.25% | 0.75% | max 0.040% | max 0.040% |

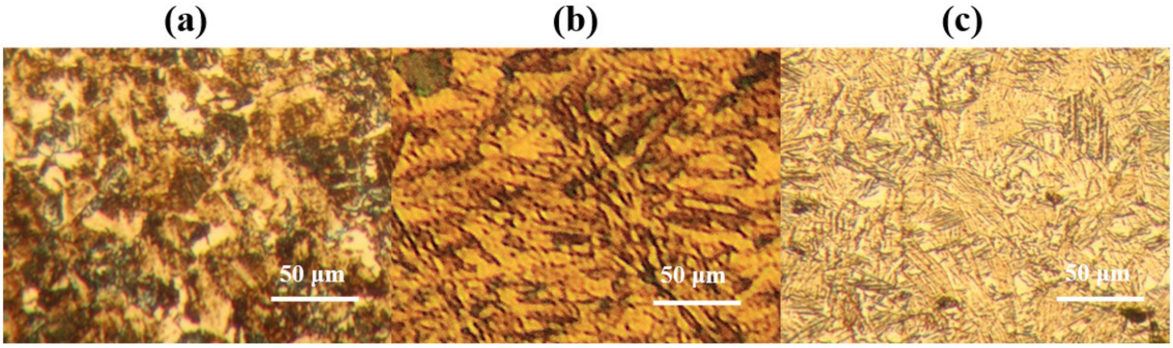


Fig. 1. Optical micrographs of: a)pearlite, b)martensite and c)bainite structures for CK45 steel.

When the specimens were held in the furnace (815ºC for 1h), their surfaces are starting to oxidation, and a film of oxide formed on the samples' surfaces, as shown in Fig 2. Since austenite grain boundaries have a greater chemical potential than other parts of the surface, they are more oxidate. The oxidation method depends on the fact that when steels are heated in an oxidizing atmosphere, oxidation takes place in part preferentially along the grain boundaries, therefore the grain boundaries are revealed by oxide accumulation. Thus before inspecting to remove this thin film, the surfaces slightly were sanded with 2000 sandpaper and then polished with alumina particles. With etching austenite, grain boundaries and structure of steels were revealed better.

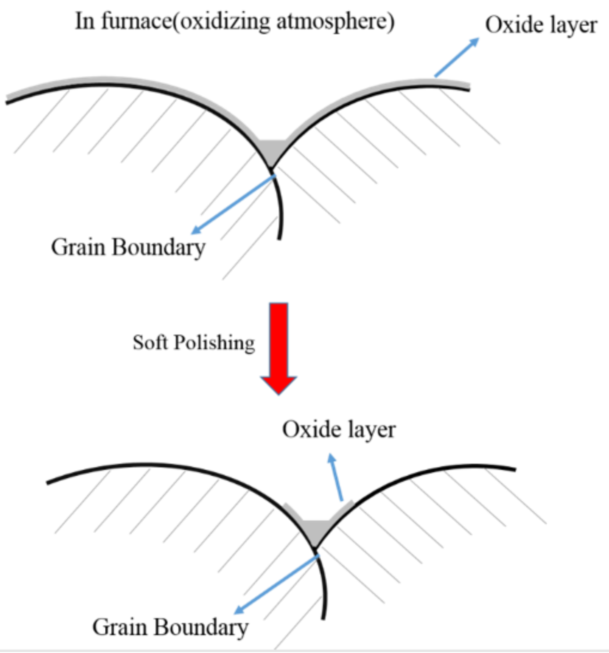


Fig. 2. Schematic of prior austenite grain boundary detection using oxidation method.

Fig 3 shows images obtained for martensitic CK45 steel heat treated at 850ºC for 1h. At low magnification, images in Fig. 3a and more magnification (Fig. 3b) show a network of grain boundaries clearly. Fig. 4a shows the results obtained for the bainite specimen after heat treatment and etching and Fig. 4b shows a higher magnification image of the microstructure for this specimen and in both images, austenite grain boundaries have been seen truly. The austenite grain boundaries and microstructure of the specimen with the ferrite-perlite structure are shown in Fig. 5(a,b). Fig. 5b shows a higher magnification image, as shown, light areas represent ferrite and dark areas represent pearlite structure. From these results, it is obvious that using the applied method in this study is extremely useful for revealing the austenite grain boundaries in a different micro-structure of CK45 steels.

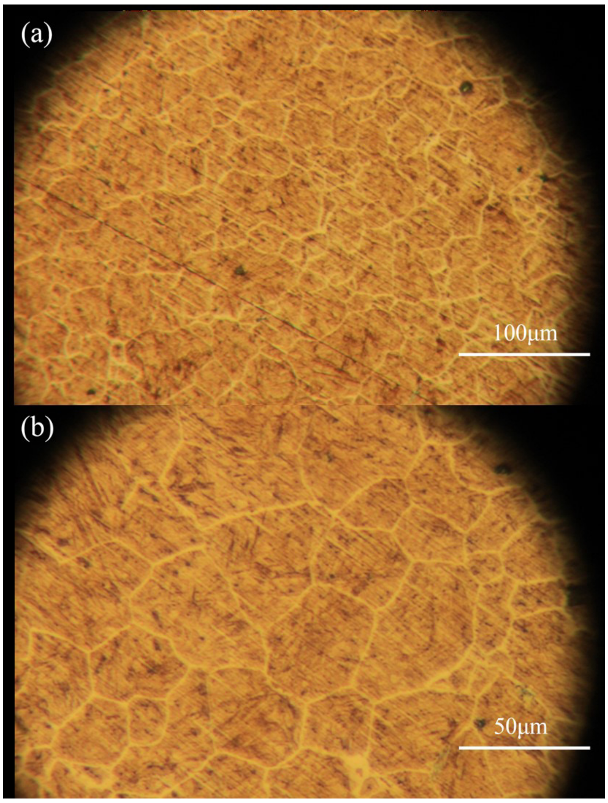


Fig. 3. Optical micrographs of the austenite grains and martensitic structure, a)200x, b)400x.

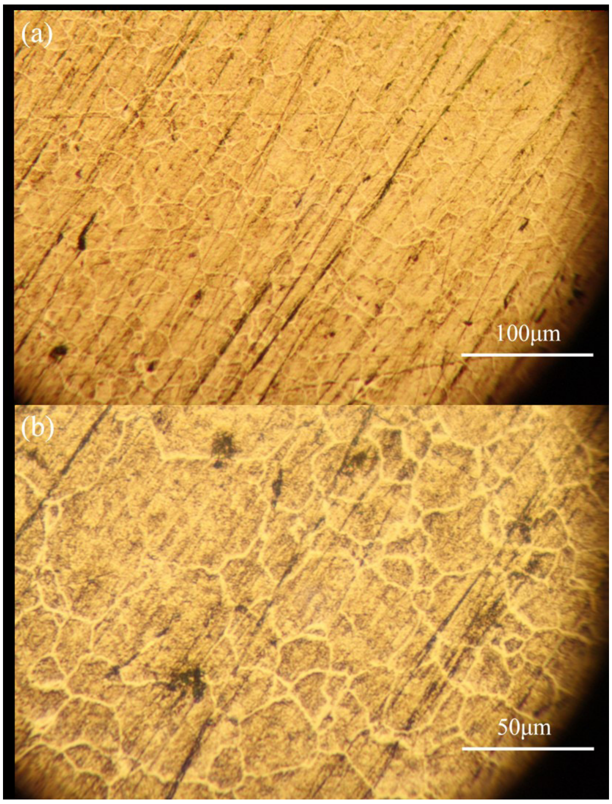


Fig. 4. Optical micrographs of the austenite grains and bainite structure, a)200x, b)400x.

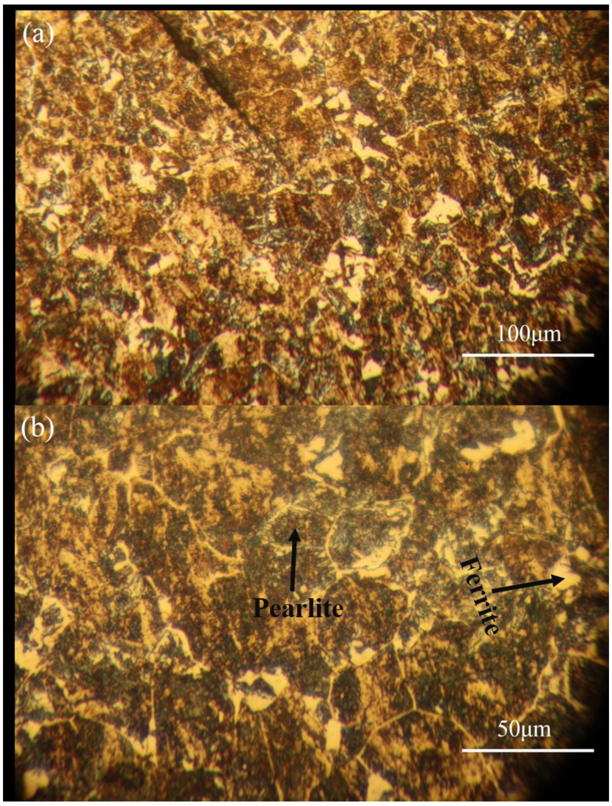


Fig. 5. Optical micrographs of the austenite grains and ferrite-perlite structure, a)200x, b)400x.

## 4. Conclusions

Revealing the austenite grain boundaries is an important step in the characterization of steel. We first used an etchant based on picric acid and a wetting agent to reveal the prior-austenite grain boundary of CK45 steel with three structures of perlite, bainite, and martensite; but the results of optical microscopy showed that the chemical etching solution used did not show sensitivity to the appearance of prior-austenite grain boundaries. Therefore, we used a two-stage method of heat treatment (oxidation) and then etching to reveal the boundaries of the prior austenite grains. Prior-austenite grain boundaries of three structures of CK45 steel (ferrite-perlite, bainite, martensite) were well revealed.

## References


[1] G. E. TOTTEN: Steel heat treatment: metallurgy and technologies, CRC press, 2006, 17.
[2] R. Singh: Applied Welding Engineering: Processes, Codes, and Standards, Butterworth-Heinemann, Oxford, UK, (2020).
[3] M. Rafati, A. Mostafapour, H. Laieghi, M. Chandra Somani, J. Kömi: Mater., 14.15 (2021), 4098.
[4] K. D. Selman: Engineering and technology journal., 29(8) (2011), 1610-1618.
[5] D. San Martin., P.E.J. Rivera Diaz Del Castillo, E. Peekstok, S. Zwaag: Mater. Charact., 58 (2007), 455.
[6] Y. Prawoto, N. Jasmawati, K. Sumeru: Mater. Sci. Eng., 28.5 (2012), 461.
[7] H. Ohtani, F. Terasaki, T. Kunitake: Tetsu to Hagané., 58.3 (1972), 434-451.
[8] NJ. Petch: Journal of the Iron and Steel institute., 174 (1953), 25.
[9] A. Grange: Strengthening steel by austenite grain refinement, ASM Trans Quart, 59.1 (1966), 26.
[10] D. San Martin, Y. Palizdar, R.C. Cochrane, R. Brydson,; A.J. Scott: Mater. Charact., 61 (2010), 584.
[11] American Society for Testing and Materials, Book of ASTM Standards Part 31, E-112-63, Standard Methods of Estimating the Average Grain Size of Metals, Philadelphia, American Society for Testing and Materials, (1969), 444.
[12] GF. Vander Voort: Metallography: principles and practice, McGraw-Hill, New York, (1984), 219.
[13] H. Modin, S.Modin: Metallurgical microscopy. Elsevier, (2016).
[14] E. Schacht, J. Richter: P, M., 35 (1998), 384.
[15] JR. Vilella: Metallographic technique for steel, OH: American Society for Metals, Metals Park, Cleveland, (1938), 26.
[16] G. Krauss: Steels: heat treatment and processing principles. Materials Park, OH: ASM International, (1988), 188.
[17] GF. Vander Voort: Metallography: principles and practice McGraw-Hill, New York, (1984), 322.
[18] WI. Halliday: ISI Special Report., 81 (1963), 65.
[19] M.W. Lui, I. LE MAY: Mater. Charact., 4 (1971), 443.
[20] P. Baldinger, G. Posch, and A. Kneisslaas: P. M., 31 (1994), 252.