

Research Article

Identification of Bainite and Martensite Phases in Cold Work Tool Steel D3 Using Color Metallography

Adnan Alizadeh Naeini* , Seyed Sadegh Ghasemi Banadkouki ,
Amirhossein Mehrbani, Hamidreza Karimi Zarchi

Faculty of Mining and Metallurgical Engineering, Yazd University, Yazd, Iran

Abstract

Background: AISI D3 cold-work tool steel is a high-carbon, high-chromium alloy renowned for its exceptional wear resistance and hardness, making it crucial for applications such as dies and cutting tools. The performance of this steel is intrinsically linked to its microstructure, which, depending on the heat treatment, can be a complex mixture of primary carbides, martensite, bainite, and pearlite. Accurate identification and differentiation of these phases are essential for quality control and predicting material behavior. **Objective:** This research aims to systematically evaluate the efficacy of various color metallography techniques for the clear identification and differentiation of bainite, martensite, and other micro-constituents in annealed D3 tool steel. This study highlights a cost-effective alternative to more advanced and expensive characterization methods like electron microscopy. **Methods:** Samples of D3 steel were subjected to an annealing heat treatment cycle, involving austenitizing at 1000 °C followed by slow furnace cooling, to generate a multi-phase microstructure. Standard metallographic preparation was followed by the application of several chemical etchants. Single-stage etching techniques using Nital, Vilella, Sodium Metabisulfite, and Marshall's reagent were employed, alongside two-stage techniques combining Nital with Marble's reagent and Nital with Sodium Metabisulfite. The resulting microstructures were analyzed using optical microscopy. Furthermore, quantitative phase analysis was performed using image analysis software to determine the volume fraction of each constituent. **Results:** The findings indicated that while single-stage etching with Nital or Vilella could identify carbides, they failed to distinguish between bainite and martensite. The two-stage technique using Nital followed by Marble's reagent provided excellent differentiation between carbides, martensite (light brown), and bainite (bright green). However, the most effective overall technique was single-stage etching with aqueous sodium metabisulfite, which successfully revealed all micro-constituents-carbides, martensite, bainite, and pearlite-simultaneously with superior contrast and clarity. Quantitative analysis revealed a microstructure composed of approximately 18% carbides, 55% martensite, 22% bainite, and 5% pearlite. Color metallography, particularly using sodium metabisulfite, proves to be a highly effective, rapid, and economical method for the comprehensive microstructural analysis of D3 tool steel. The developed techniques provide a reliable tool for phase identification in high-chromium tool steels, facilitating process control and material development.

Keywords

Cold Work Tool Steel D3, Phase Differentiation, Martensite, Bainite, Color Metallography, Quantitative Analysis

1. Introduction

*Corresponding author: alizade.adnan@yahoo.com (Adnan Alizadeh Naeini)

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Cold-work tool steels are a critical class of materials used for shaping, cutting, and forming other materials at ambient temperatures. Among these, high-carbon, high-chromium steels, specifically the AISI D-series, are distinguished by their exceptional properties. D3 tool steel (also known as DIN 1.2080), with its high carbon (around 2.1%) and chromium (around 12%) content, exhibits outstanding wear resistance, high compressive strength, and good dimensional stability after heat treatment [10]. These characteristics make it an ideal choice for manufacturing forming rolls, drawing dies, and powder compaction tooling.

The mechanical properties of D3 steel are not solely dependent on its chemical composition but are critically dictated by its microstructure [8]. The applied heat treatment process governs the final phase constitution, which can be a complex mixture of a martensitic or bainitic matrix, large primary chromium-rich carbides, and potentially fine secondary carbides and retained austenite. The size, morphology, and distribution of these phases, particularly the hard carbides and the matrix phases of martensite and bainite, determine the steel's ultimate hardness, toughness, and in-service performance. Therefore, the ability to accurately identify and quantify these microstructural constituents is not merely an academic exercise but a fundamental requirement for industrial quality control, failure analysis, and the development of optimized heat treatment cycles.

While advanced techniques such as Scanning Electron Microscopy (SEM) and Electron Backscatter Diffraction (EBSD) offer high-resolution analysis, they are often time-consuming, expensive, and require specialized equipment and expertise [1]. In contrast, color metallography presents a powerful, rapid, and cost-effective alternative for phase identification [2]. This technique utilizes chemical etchants that react differently with various phases, depositing thin interference films on the specimen surface. The thickness of these films varies depending on the electrochemical potential and crystallographic orientation of the underlying phase, resulting in distinct colors under an optical microscope. This color contrast greatly enhances the ability to distinguish between phases that may appear similar in conventional black-and-white microscopy, such as bainite and tempered martensite [3].

Despite its advantages, the application of color metallography requires careful selection of etchants and procedures, as their effectiveness can vary significantly with alloy composition and microstructure. In this research, we undertake a systematic investigation to identify the most effective etching solutions—both single-stage and two-stage—for differentiating the bainite, martensite, pearlite, and carbide phases in annealed D3 cold-work tool steel. The aim is to establish a reliable and accessible methodology for the detailed microstructural characterization of this important industrial alloy.

2. Materials and Methods

2.1. Material and Heat Treatment

In this study, samples of AISI D3 (DIN 1.2080) cold-work tool steel were utilized. The material was sourced from a 16mm diameter rod, and its chemical composition, as determined by quantitative analysis, is presented in Table 1. The samples were machined into cylindrical shapes with a height of 10mm.

To create a multi-phase microstructure containing pearlite, bainite, martensite, and carbide phases, the samples were subjected to a full annealing cycle. This process involved austenitizing the samples in a furnace at a temperature of 1000 °C for 1 hour to ensure complete dissolution of secondary carbides and homogenization of the austenite. Subsequently, the samples were cooled slowly inside the furnace over a period of 8 hours to room temperature. This slow cooling rate, as informed by the Continuous Cooling Transformation (CCT) diagram for this steel, allows for sequential transformation of austenite into pearlite, bainite, and finally martensite. A schematic of the heat treatment cycle is shown in Figure 1.

2.2. Metallographic Preparation and Etching

For microstructural investigation, the heat-treated samples were prepared using standard metallographic procedures. The samples were ground using a series of silicon carbide papers and then polished to a mirror finish using 0.3 µm alumina slurry and detergent.

To reveal the microstructure, a variety of chemical etching solutions were employed. These included single-stage etchants such as 4% Nital, Picral, Vilella, Sodium Metabisulfite, Marble's, Marshall's, Klemm's, and Ferric Chloride. Additionally, two-stage etching procedures were tested, specifically using 4% Nital followed by Sodium Metabisulfite, and 4% Nital followed by Marble's reagent. The chemical compositions of the primary etchants used for analysis are listed in Table 2. The etched microstructures were observed using an Olympus PMG3 optical microscope, and micrographs were captured at various magnifications with a JVC TK-C1380E camera.

2.3. Quantitative Phase Analysis

To supplement the qualitative observations, quantitative analysis was performed on the most clearly resolved micrograph to determine the volume fraction of the constituent phases. The micrograph obtained using the sodium metabisulfite etchant (Figure 3e) was selected for this analysis due to its superior contrast among all phases. The analysis was conducted using the open-source image analysis software ImageJ [4]. The procedure involved the following steps:

- 1) *Scale Calibration*: The image scale was set based on the magnification bar.
- 2) *Color Thresholding*: The "Color Threshold" tool was used to segment the image based on color. Distinct col-

or ranges corresponding to each phase (carbides: white; martensite: brown; bainite: dark green/blue; pearlite: layered black/white) were selected [5].

- 3) *Area Fraction Measurement*: The "Analyze Particles" function was used to calculate the total area of the pixels selected for each phase. The area fraction (%) for each phase was then determined relative to the total image area. This process was repeated for each distinct phase in the microstructure [21].

3. Results and Discussion

3.1. Microstructural Characterization

The chemical composition of the D3 steel used in this study is detailed in Table 1. The high concentrations of carbon (2.10%) and chromium (11.2%) are responsible for the formation of a large volume fraction of hard, wear-resistant chromium carbides and contribute to the steel's exceptionally high hardenability.

Table 1. Quantitative analysis of the chemical composition of the D3 cold-work tool steel rod used.

Element	C	Cr	Mn	Si	S	P	Mo	Ni	V	Fe
Weight %	2.10	11.2	0.255	0.195	0.025	0.026	0.088	0.197	0.052	balance

Table 2. Chemical composition of the primary etchant solutions used in this study.

Etchant Solution Name	Chemical Composition
Nital	2–4% HNO ₃ +96–98% C ₂ H ₅ OH
Vilella	1g picric acid (C ₆ H ₃ N ₃ O ₇)+5ml HCl+95ml C ₂ H ₅ OH
Marble's	20g CuSO ₄ +100ml HCl+100ml H ₂ O
Sodium Metabisulfite	10–12g Na ₂ S ₂ O ₅ +100ml H ₂ O
LePera	50ml Na ₂ S ₂ O ₅ solution+50ml Picral solution
Marshall's	80ml H ₂ O+g Oxalic acid (C ₂ H ₂ O ₄) +4ml H ₂ O ₂

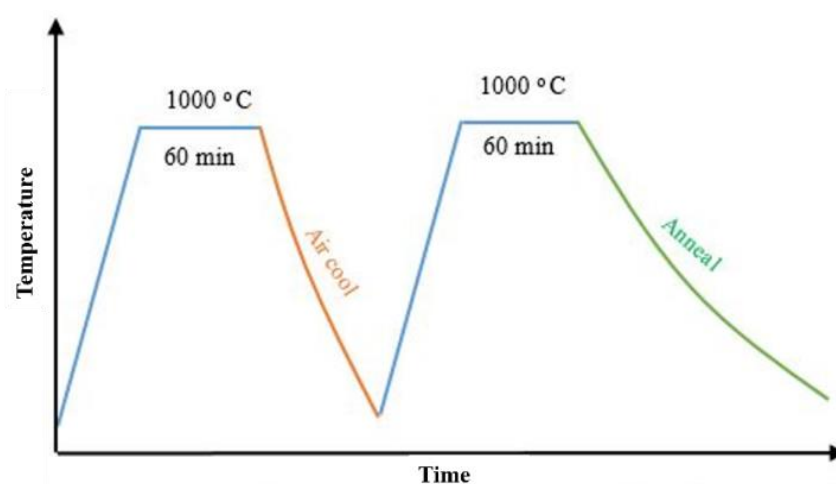


Figure 1. Schematic of the annealing heat treatment cycle performed on the D3 steel samples.

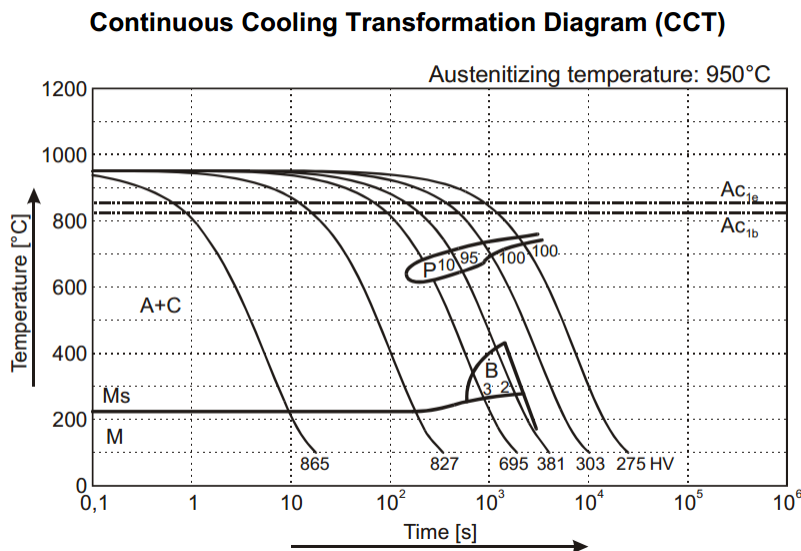


Figure 2. Continuous Cooling Transformation (CCT) diagram for D3 tool steel, illustrating the transformation regions for pearlite (P), bainite (B), and martensite (M).

The annealing heat treatment (Figure 1) was designed based on the CCT diagram for D3 steel (Figure 2). The slow cooling path ensures that the austenite transforms sequentially across different temperature ranges, resulting in a mixture of pearlite, bainite, and martensite, embedded with pri-

mary carbides that remain undissolved at the austenitizing temperature.

The microstructures revealed by various etchants are shown in Figure 3. A summary of the effectiveness of each etching technique is provided in Table 3.

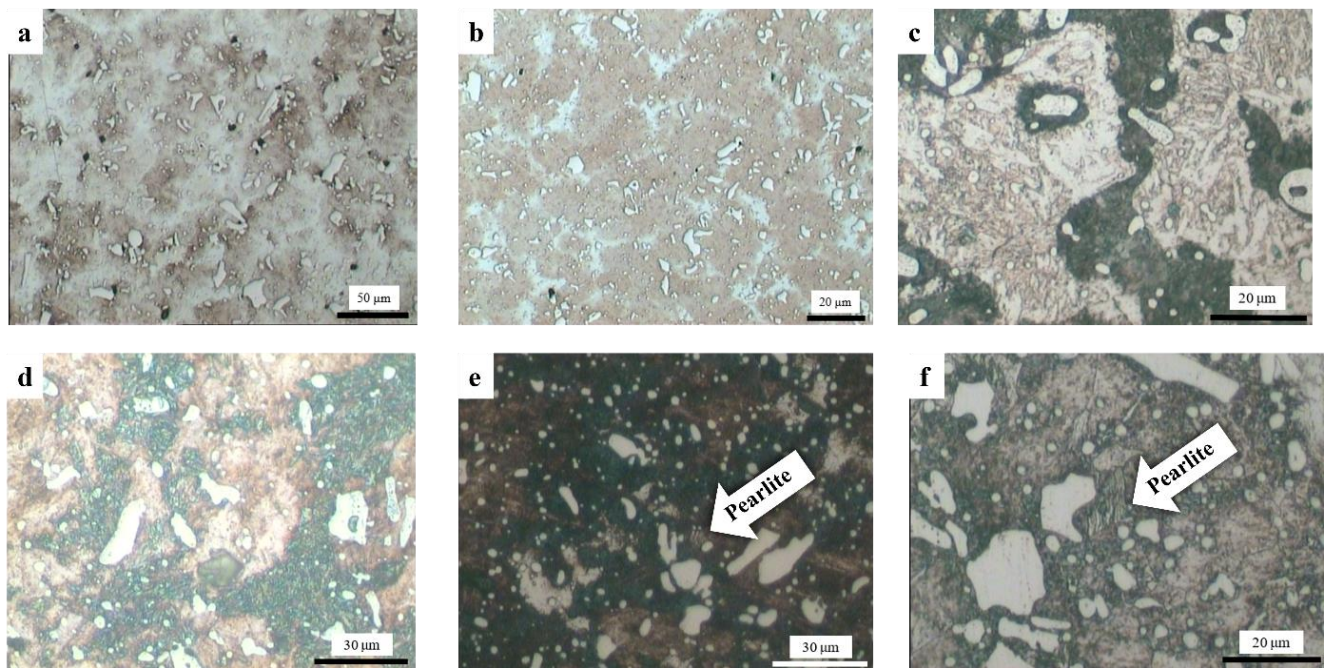


Figure 3. Optical micrographs of the annealed D3 steel sample etched with various reagents, showing the differentiation of phases.

Table 3. Summary of Etching Results and Phase Differentiation Effectiveness.

Etching Technique	Phases Revealed Clearly	Phases Not Differentiated	Overall Contrast/Clarity
Nital (single-stage)	Carbides, Martensite Matrix	Bainite, Pearlite	Poor

Etching Technique	Phases Revealed Clearly	Phases Not Differentiated	Overall Contrast/Clarity
Vilella (single-stage)	Carbides, Martensite Matrix	Bainite, Pearlite	Poor
Nital + Na-metabisulfite	Martensite (light brown), Bainite (dark green)	Pearlite, Carbides (obscured)	Moderate
Nital + Marble's	Carbides, Martensite (light brown), Bainite (bright green)	Pearlite	Good
Sodium Metabisulfite (single-stage)	Carbides, Martensite, Bainite, Pearlite	None	Excellent
Marshall's (single-stage)	Carbides, Martensite, Bainite, Pearlite	None	Good

1) *Single-Stage Etching (Nital and Vilella)*: As seen in [Figure 3a and 3b](#), both 4% Nital and Vilella's reagent were effective at revealing the primary chromium carbides (bright white particles) by attacking their boundaries. However, both etchants colored the matrix a uniform brownish tint, failing to provide any contrast between the martensite, bainite, and pearlite phases. This limitation is common for general-purpose attack etchants when dealing with complex, multi-phase microstructures [6-9].

2) *Two-Stage Etching*:

- i. *Nital + Sodium Metabisulfite*: This combination ([Figure 3c](#)) produced distinct colors for the matrix phases, with martensite appearing light brown and bainite appearing dark green. This demonstrates a clear improvement in differentiating the acicular structures. However, this technique obscured the carbide boundaries, making them difficult to resolve clearly. The pearlitic constituent was also not revealed.
- ii. *Nital + Marble's Reagent*: This two-step process ([Figure 3d](#)) offered excellent clarity. The primary carbides remained bright white and sharply defined. The martensitic matrix was colored light brown, while the bainite was tinted a distinct bright green. This method provides strong, reliable differentiation for carbides, martensite, and bainite. Its only drawback was the failure to

reveal the fine pearlitic colonies.

3) *Single-Stage Tint Etching (Sodium Metabisulfite and Marshall's)*:

- i. *Sodium Metabisulfite*: As shown in [Figure 3e](#), a single immersion in 10% aqueous sodium metabisulfite yielded the most comprehensive results. This etchant successfully differentiated all phases with excellent contrast: carbides appeared bright white, martensite was colored brown, bainite was a dark green/blue, and, upon closer inspection, the fine lamellar structure of pearlite was also clearly visible. The clarity and distinction between all phases were superior to all other methods tested.
- ii. *Marshall's Reagent*: This etchant ([Figure 3f](#)) produced results similar to sodium metabisulfite, revealing all four micro-constituents. However, the color contrast, particularly between the matrix phases, was less pronounced compared to that achieved with sodium metabisulfite.

3.2. Quantitative Phase Analysis

The volume fractions of the microstructural constituents were quantified from the micrograph etched with sodium metabisulfite ([Figure 3e](#)) using ImageJ software. The results, summarized in [Table 4](#), provide a quantitative description of the annealed microstructure.

Table 4. Quantitative Phase Fractions Determined by Image Analysis.

Microstructural Constituent	Color in Micrograph (Figure 3e)	Volume Fraction (%)
Primary Carbides	Bright White	18.3 ± 1.5
Martensite	Brown	54.8 ± 2.1
Bainite	Dark Green / Blue	21.7 ± 1.8
Pearlite	Lamellar Black/White	5.2 ± 0.9

The analysis shows that the microstructure is predominantly martensitic, which is expected given the high harden-

ability of D3 steel, even with slow furnace cooling. The significant fraction of undissolved primary carbides is a key

feature of this steel, contributing directly to its high wear resistance. The presence of bainite and a small amount of pearlite confirms that the cooling rate was slow enough to allow for diffusional transformations to occur before the martensite start (Ms) temperature was reached.

3.3. Discussion

The results clearly demonstrate the superiority of color etching over conventional attack etching for analyzing complex, multi-phase steel microstructures. The mechanism behind color or "tint" etching involves the chemical deposition of a thin, transparent film on the specimen's surface [7]. For etchants like sodium metabisulfite or Marble's reagent, this is often a sulfide or oxide film. The color observed arises from light interference between rays reflecting from the top surface of the film and those reflecting from the film-metal interface. The resulting color is highly dependent on the film's thickness, which is a function of the electrochemical potential and crystallographic orientation of the underlying phase [10-15]. Martensite, with its highly strained body-centered tetragonal (BCT) lattice, reacts at a different rate than the less-strained body-centered cubic (BCC) structures of bainite and ferrite, leading to different film thicknesses and thus different colors [16]. This explains why sodium metabisulfite, a well-regarded tint etchant for multiphase steels [3, 17], could successfully differentiate martensite (brown) from bainite (dark green/blue).

The success of the two-stage Nital + Marble's technique also aligns with findings in the literature for other advanced high-strength steels, where multi-step etching is often necessary to reveal all constituents [19]. The initial Nital etch likely creates subtle surface relief and activates the surface, allowing the subsequent Marble's reagent to deposit its copper-based film more selectively on the bainitic and martensitic phases. Marble's reagent is known to function via a displacement reaction where copper ions (Cu^{2+}) in the solution plate onto the more active (less noble) phases of the steel, creating the colored film [18-21].

The quantitative results underscore the structure-property relationship in D3 steel. The high volume fraction of large primary carbides (~18%) is the primary source of the steel's exceptional abrasive wear resistance [22-24]. The predominantly hard martensitic matrix (~55%) provides high compressive strength and hardness, while the presence of bainite (~22%) can contribute to a modest improvement in toughness compared to a fully martensitic structure [25-28]. The ability to quantify these phases using a simple and accessible technique like the one demonstrated here is therefore of significant practical value for predicting material performance.

This study focused on an annealed condition. A valuable direction for future work would be to apply these validated etching techniques to D3 steel subjected to different heat treatments, such as conventional quench-and-temper cycles or deep cryogenic treatment (DCT). DCT is known to pro-

mote the transformation of retained austenite and encourage the precipitation of finer, more uniformly distributed secondary carbides, further enhancing wear resistance and hardness [29-31]. The color etching methods identified here would be an excellent tool for characterizing these subtle but critical microstructural changes [32-36].

4. Conclusion

This study systematically evaluated various chemical etching techniques for the microstructural characterization of annealed AISI D3 cold-work tool steel. The investigation yielded the following key conclusions:

- 1) Conventional single-stage etchants like Nital and Vilella are insufficient for analyzing the complex microstructure of D3 steel, as they fail to differentiate between bainite, martensite, and pearlite.
- 2) A two-stage etching technique using 4% Nital followed by Marble's reagent provides excellent, clear differentiation of primary carbides, martensite, and bainite, making it a highly reliable method for qualitative analysis of these specific phases.
- 3) The most effective and comprehensive method identified was single-stage etching with a 10% aqueous solution of sodium metabisulfite. This simple immersion technique successfully revealed and distinguished all four primary micro-constituents-carbides, martensite, bainite, and pearlite-with superior color contrast.
- 4) Quantitative image analysis, enabled by the clear phase segmentation from the sodium metabisulfite etch, determined the microstructure to consist of approximately 18% carbides, 55% martensite, 22% bainite, and 5% pearlite.
- 5) The findings confirm that color metallography is a powerful, economical, and efficient tool for the detailed analysis of high-carbon, high-chromium tool steels, providing crucial information for quality control and material research.

Abbreviations

CCT Continuous Cooling Transformation

Author Contributions

Adnan Alizadeh Naeini: Conceptualization, Data curation, Methodology, Writing - review & editing

Seyed Sadegh Ghasemi Banadkouki: Supervision, Writing - original draft

Amirhossein Mehrbani: Investigation

Conflicts of Interest

The authors declare no conflicts of interest.

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