

A Study on Heat Treatment Processes for Cold-Heading Steel

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1. Foreword

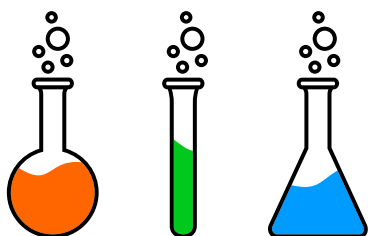
To achieve a 90% tempered sorbite or 90% tempered troostite microstructure with good overall mechanical properties through heat treatment (normalizing) of high-strength bolts, the material used must have sufficient hardenability to ensure that the core of the fastener's threaded cross-section is in a "hardened state" and approximately 90% martensitic structure in the threaded cross-section of the fastener prior to tempering. Therefore, cold-heading steel wire must possess high performance characteristics. Hardenability refers to the maximum hardness achieved by steel after quenching; it primarily depends on the carbon content of the steel. Hardenability is one of the key process properties of steel, directly affecting the mechanical properties and service life of cold-heading steel.

The hardenability of steel refers to its ability to develop a martensitic microstructure under specific cooling conditions following an austenitic transformation. The degree of hardenability can be demonstrated by the hardness distribution in the hardenability layer formed during the austenitization process of the steel under specified conditions. For example, the primary role of boron (B) in steel is to increase its hardenability, thereby reducing the need for other relatively scarce and expensive metals such as Ni, Cr, and Mo. A trace amount of boron (0.0008%) can significantly improve the steel's hardenability, but an excess of boron (0.005%) offers no further benefit. This effect is highly sensitive to the austenitizing temperature; temperatures that are too high or too low cannot effectively improve the steel's hardenability.



Generally, Grade 8.8 applications use 10B21 and ML20MnTiB steels, while Grade 10.9 applications use ML40Cr and ML35CrMo steels, among other materials. Both 10B21 and ML20MnTiB steels are low-carbon cold-heading steels; ML40Cr and ML35CrMo are both medium-carbon cold-heading steels. During quenching, the former is generally quenched in a PAG aqueous solution, which has a relatively fast cooling rate, while the latter is typically quenched in rapid-quenching oil, which has a slower cooling rate. Therefore, it is of great significance to study the properties of both materials to determine which is more in line with industry trends.

2. Experiments on Heat Treatment Processes for Cold-Heading Steel



Taking the boron-containing cold-heading steels 10B21 and ML20MnTiB as examples, this study investigates the effect of different austenitizing temperatures on hardenability. For the process tests on both 10B21 and ML20MnTiB steels, a PAG aqueous solution quenching medium was used. Using a thermal expansion phase transformation analyzer, the CCT curves of the materials were determined to clarify the effect of cooling rates on microstructural transformation.

The tests utilized $\phi 24$ mm diameter wire rods of 10B21 and ML20MnTiB steel; their measured chemical compositions are shown in **Table 1**. Based on the chemical composition of the steels, the CCT curves for both steels were calculated using JMatPro (see **Figure 1**), revealing that the

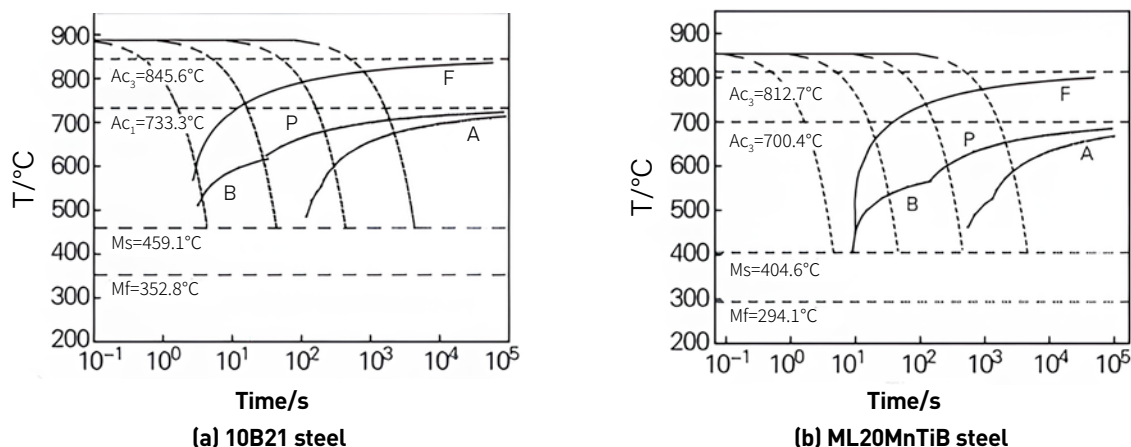


critical points Ac_3 for 10B21 and ML20MnTiB steels were 846°C and 813°C, respectively. Next, a Vickers microhardness tester was used to measure the microhardness of the materials. During the testing process, the tester's load was consistently maintained at 980 N to maximize the accuracy of the comparisons and ensure more meaningful practical results.

▼ Table 1. Chemical Composition of 10B21 and ML20MnTiB Steels (Mass Fraction, %)

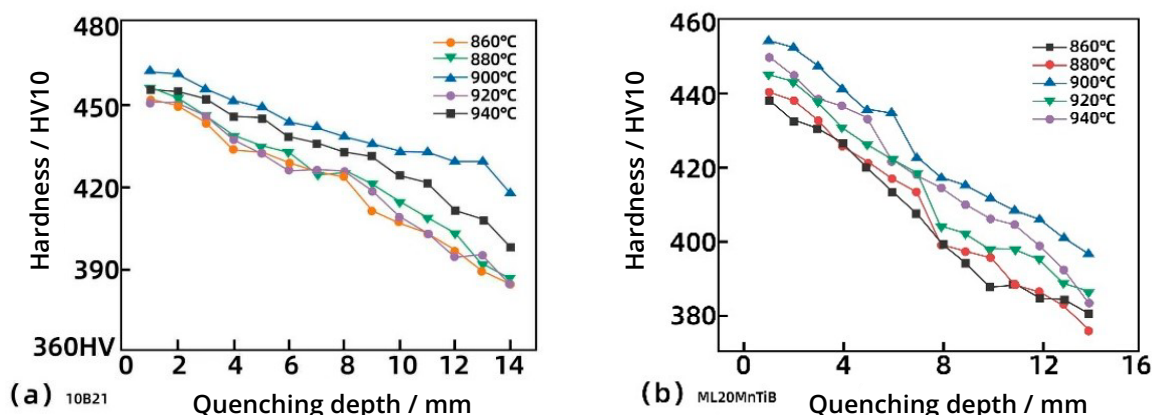
Steel Grade	C	Si	Mn	P	S	Ala	Ti	B
10B21	0.215	0.14	0.78	0.015	0.004	0.031	0.036	0.0020
ML20MnTiB	0.205	0.23	1.49	0.012	0.001	0.028	0.057	0.0027

▼ Figure 1. CCT Curves for 10B21 and ML20MnTiB Steels



The test quenching temperatures ranged from 860 to 940 °C. A total of five sets of specimens were tested at 860, 880, 900, 920, and 940 °C. For each set, eight specimens were tested, with hardness measurements taken at 3 mm intervals from the edge to the center. The hardness distributions of the 10B21 and ML20MnTiB steels are shown in **Figure 2**; Based on the quenching hardness distribution, the average hardness of both steels gradually decreases with increasing depth. During austenitization at 860–940 °C, the hardness of 10B21 steel ranged from 375 to 454 HV10, while that of ML20MnTiB steel ranged from 386 to 468 HV10. Both steels exhibited their highest overall hardness during austenitization at 900 °C.

▼ Figure 2. Hardness Gradients of 10B21 Steel (a) and ML20MnTiB Steel (b) at Different Austenitizing Temperatures



3. Validation of Heat Treatment Processes for 10B21 and ML40Cr Steel

The quenching temperature is generally selected as $Ac_3 + (30-50) ^\circ C$. The Ac_3 value for 10B21 steel is 846 °C, and for ML40Cr steel it is 805 °C. Therefore, the quenching temperature for 10B21 steel is $\geq 890 ^\circ C$, and for ML40Cr steel it is $\geq 860 ^\circ C$.

3.1. Test Specimens

A test set consists of 8 hardness specimens, 5 tensile specimens, and 3 impact specimens. Specimens of two different materials are prepared, and tests with different parameters are conducted. The hardness testing instrument is the HR-150DT digital Rockwell hardness tester; the tensile testing equipment is the CHT4605 CMT microcomputer-controlled electro-hydraulic servo universal testing machine; the impact testing equipment is the JB-300B low-temperature impact testing machine, with test specimens measuring 55 mm × 10 mm × 10 mm with a V-notch; A DMI3000M metallographic microscope was used for microstructural observation.

3.2. Test Plan

The target internal mechanical properties for high-strength bolts, as specified in GB/T 3098.1, are shown in **Table 2**. Test plans for 10B21 steel and ML40Cr steel were developed separately based on the quenching medium, quenching temperature, and specimen type, as shown in **Table 3**.

▼ Table 2. Target Internal Mechanical Properties for High-Strength Bolts

Performance Metrics	Rm Tensile Strength /MPa	ReL Yield Strength/ MPa	Rockwell Hardness / HRC	-20°C(Sub-zero) KV2 Low-Temperature Impact Energy /J
Gr. 8.8	870~950	≥660	26.5~31.0	≥27
Gr. 10.9	1080~1140	≥960	35~37.5	≥27

▼ Table 3. Heat Treatment Process Validation Plan for 10B21 and ML40Cr Steels

Bolt Material	Grade	Quenching Process	Tempering Process	Tensile Test Specimen	Impact Test Specimen
10B21 Steel	Gr. 8.8≤M10	880°C×80min, fast oil	440~460°C×90min	5 of each, for a total of 15	3 of each, for a total of 9
	Gr. 8.8 M12	890°C×80min, fast oil	440~460°C×90min		
	Gr. 8.8 M16	880°C×90min, 3%~3.5% aqueous solution	450~470°C×90min		
ML40Cr Steel	Gr.10.9≤M12	870°C×90min, fast oil	490~500°C×90min	5 of each, for a total of 15	3 of each, for a total of 9
	Gr.10.9 M16	880°C×110min, fast oil	500~510°C×90min		
	Gr.10.9 M30	850°C×135min, 7%~8.5% aqueous solution	510~520°C×90min		

3.3 Validation Results

3.3.1 Quenching, Tempering Hardness

The measured hardness of 10B21 steel after quenching was 43.8–44.8 HRC, which corresponds to the hardness value achieved when approximately 90% of the microstructure is martensite. After quenching in an aqueous solution at 880°C, the maximum hardness was 44.5–45.2 HRC, slightly higher than the hardness achieved with fast oil quenching.

The hardness of bolts of different specifications after tempering meets the requirements for Grade 8.8. After quenching in an aqueous solution, the tempering temperature was 10°C higher, resulting in an average hardness of 30.2 HRC; for oil quenching, both specifications were close, at 29.5 HRC and 28.9 HRC, respectively; The measured quenching hardness of ML40Cr steel was 52.8–53.3 HRC, consistent with the hardness values achieved when approximately 90% martensitic structure is formed.

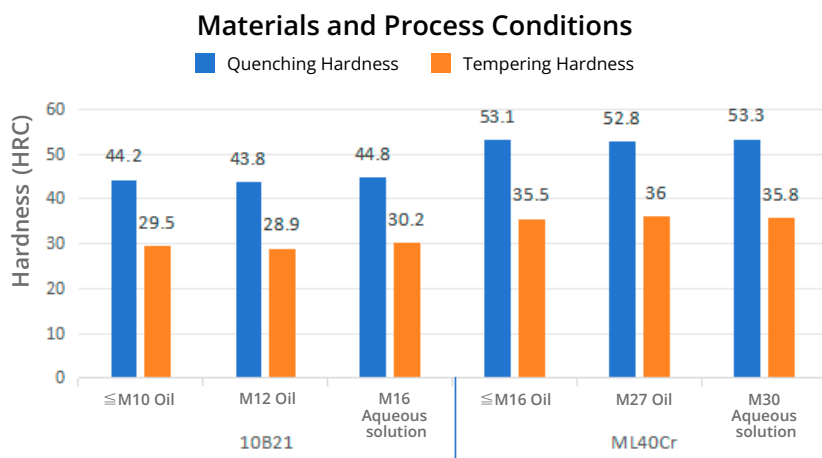
After quenching in an aqueous solution at 850°C, the maximum hardness ranged from 51.5 to 55.2 HRC, which is slightly higher than the hardness achieved with fast oil quenching. The hardness of bolts of different specifications after tempering meets the requirements for Grade 10.9. After quenching in an aqueous solution, the tempering temperature was 10°C higher, resulting in an average hardness of 35.8 HRC; for oil quenching, the hardness of both specifications was similar, at 35.5 HRC and 36.0 HRC, respectively. The quenching and tempering hardness values are shown in **Figure 3**.

3.3.2 Tensile Strength, Impact Resistance

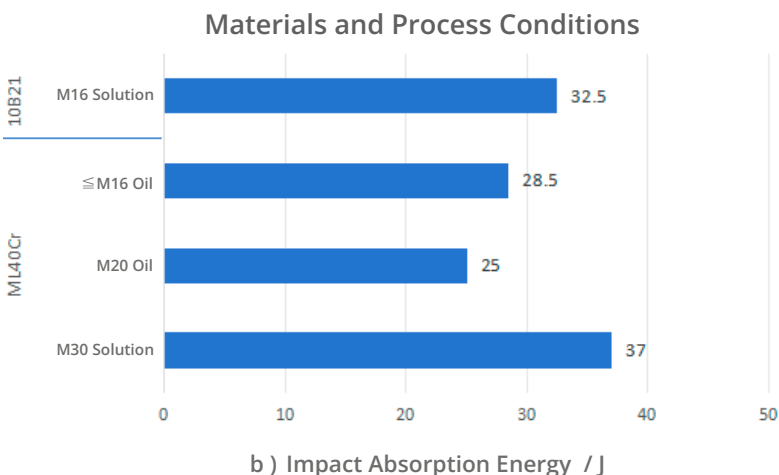
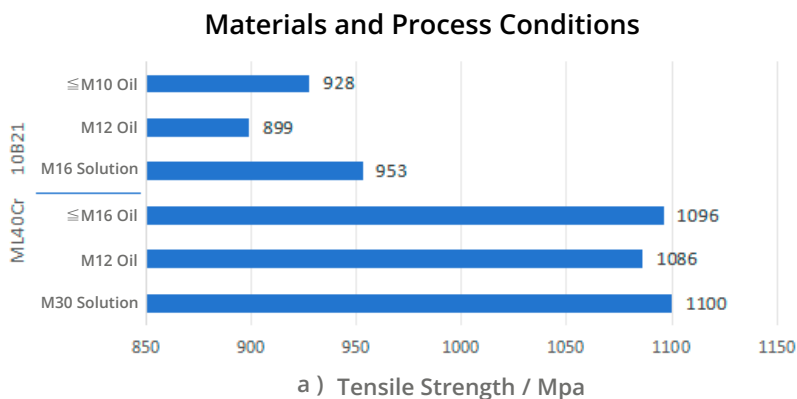
The tensile strength and impact resistance of the quenched and tempered 10B21 steel Grade 8.8 bolts and the ML40Cr steel Grade 10.9 bolt specimens are shown in **Figure 4**.



▼ Figure 3. Hardness After Quenching and Tempering for The Two Materials Under Different Processing Conditions (Fast Quenching Oil and Aqueous Solution)



▼ Figure 4. Test Results for Tensile Strength and Impact Energy Absorption of the Two Materials Under Different Processing Conditions



1. For Grade 8.8 bolts made of 10B21 steel, the tensile strength is ranked from highest to lowest as follows: 880°C M16 solution-quenched → 880°C ≤ M10 oil-quenched → 880°C M12 oil-quenched; the tensile strengths of 880°C ≤ M10 oil-quenched and 880°C M12 oil-quenched are similar; In terms of impact energy absorption, only the 880°C M16 solution-quenched sample meets the 55 mm × 10 mm × 10 mm specimen requirement, achieving 32.5 J.

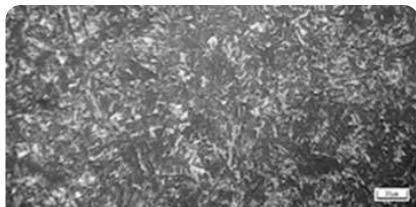
2. For Grade 10.9 bolts made of ML40Cr steel, the tensile strength ranked from highest to lowest is as follows: 850°C M30 solution-quenched → 870°C ≤ M16 oil quenched → 880°C M27 oil quenched; The impact energy absorption values, ranked from highest to lowest, are: 850°C M30 solution-quenched → 870°C ≤ M16 oil-quenched → 880°C M27 oil-quenched; The 850°C M30 solution-quenched bolts have the highest impact energy absorption at 37 J; the 880°C ≤ M16 bolts have an impact energy absorption of 28.5 J, which is basically acceptable; whereas the 880°C M27 oil-quenched bolts only reach an average of 25 J, failing to meet the standard value.

3.3.3 Metallographic Structure

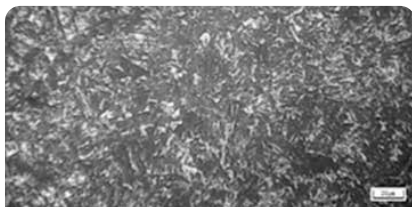
The microstructure of the specimens after quenching and tempering was examined under a 500x microscope, showing the microstructures of the two materials under different processing conditions.

10B21 steel consists of tempered troostite and ferrite. It must meet the requirements of JB/T 7293.3-2014 for inspection according to Grade 3 diagrams, with the tempered troostite microstructure rated as Grade 1–3 to be considered qualified. The metallographic structures of bolts quenched in aqueous solution at 880°C (M16), quenched in oil at 880°C (≤M10), and quenched in oil at 880°C (M12) followed by medium-temperature tempering are shown in **Figures 5 through 7**, respectively. The M16×80 bolt, solution-quenched and tempered, exhibits a structure of troostite plus a small amount of ferrite, classified as Grade 3; the M10×40 bolt, oil-quenched and tempered, exhibits a structure of troostite plus a small amount of ferrite, classified as Grade 3; and the M12×105 bolt, oil-quenched and tempered, exhibits a structure of troostite plus polygonal ferrite, classified as Grade 4. However, the microstructures reveal that the M10×40 oil-quenched bolt contains slightly more ferrite, and the M12×105 oil-quenched bolt exhibits the lowest strength and toughness. This indicates that undissolved ferrite was retained after quenching during the austenitization process at 880–890°C; In contrast, **using an aqueous solution as the quenching medium—which provides a faster cooling rate—results in a fine, uniform tempered troostite microstructure.**

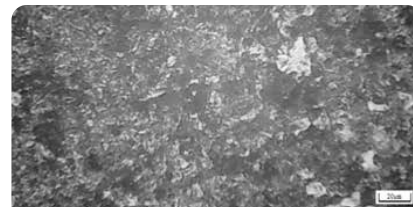
▼ Figure 5. Solution-quenched
M16×80 Bolt, Grade 3 (10B21×500)



▼ Figure 6. Oil-Quenched M10×40
Bolt, Grade 3 (10B21×500)

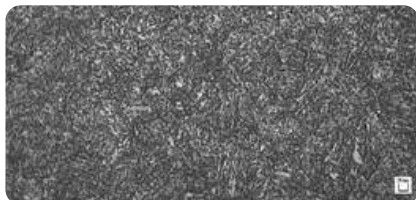


▼ Figure 7. Oil-quenched M12×105
Bolt, Grade 4 (10B21×500)



ML40Cr steel has a microstructure consisting of tempered sorbite plus a small amount of ferrite. It must comply with GB/T 38720-2020, which specifies that 90% of the microstructure must consist of tempered sorbite, and the microstructure must be evaluated as meeting Grade 1 through 6 requirements. The metallographic structures of bolts solution-quenched at 850°C (M30), oil-quenched at 870°C ($\leq$ M16), and oil-quenched and high-temperature tempered at 880°C (M27) are shown in **Figures 8 through 10**, respectively. The M30×180 bolt, solution-quenched and tempered, exhibits a sorbite structure with a small amount of ferrite (Grade 5); the M16×120 bolt, oil-quenched and tempered, exhibits a sorbite structure with a small amount of banded ferrite (Grade 6); and the M27×150 bolt, oil-quenched and tempered, exhibits a sorbite structure with prior austenitic ferrite (Grade 7). ML40Cr steel is generally quenched using fast quenching oil as the cooling medium, whereas larger-sized M30 bolts are quenched in a 7%–8.5% aqueous solution, with the temperature corresponding to the maximum cooling rate being approximately 700°C; for fast-quenching oil, the temperature corresponding to the maximum cooling rate is approximately 610°C. In other words, the temperature at which the aqueous solution quenching medium reaches the boiling stage is higher, and the duration is shorter; due to the higher cooling rate, the tempered sorbite microstructure is finer. At 870°C or higher, the ferritic microstructure in oil-quenched M16 bolts is slightly more prominent, and at 880°C, the strength and toughness of oil-quenched M27 bolts are also lower.

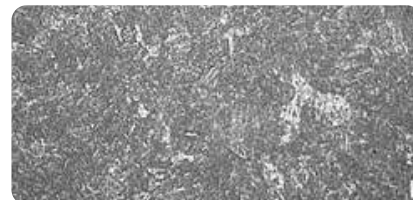
▼ Figure 8. Solution-quenched
M30×180 Bolt, Grade 5 (ML40Cr×500)



▼ Figure 9. Oil-quenched M16×120
Bolt, Grade 6 (ML40Cr×500)



▼ Figure 10. Oil-quenched M27×150
Bolt, Grade 7 (ML40Cr×500)



3.4 Insights from Mass Production of Products

As is known, heat treatment of fasteners is a technique that involves heating, holding at a specific temperature, and cooling steel using appropriate methods to achieve the desired microstructure and properties, thereby imparting ultimate performance to the material and ensuring the ultimate service life of high-strength bolts. As can be seen, the key to the quenching and cooling process lies in controlling the cooling rate. Based on the critical cooling rate, one must comprehensively consider factors such as the type of quenching medium—whether to use an aqueous solution or fast quenching oil, and prioritize factors such as the fastener's dimensions and material composition. Therefore, it is crucial to flexibly apply the CCT diagram when determining the specific quenching and cooling parameters.

For 10B21 and ML40Cr steels—the former used for Grade 8.8 bolts and the latter for Grade 10.9 bolts—only two quenching media options were considered for the tempering process: fast quenching oil and a PAG aqueous solution. For quenched and tempered 10B21 and ML40Cr steel specimens, the tensile strength and impact properties achieved with the aqueous solution quenching method were overall significantly higher than those achieved with fast quenching oil; however, there is a risk of cracking. The cost of quenching and tempering with fast quenching oil is relatively high, and the quality of the heat treatment needs to be improved. A comprehensive analysis concludes that for Grade 8.8 $\geq$ M12 10B21 steel bolts and Grade 10.9 $\geq$ M30 ML40Cr steel bolts, the most environmentally friendly and energy-efficient process involves using an aqueous solution for quenching at 880°C and 850°C, respectively, while maintaining the same tempering process. This improves mechanical properties; note that tempering must be performed promptly after quenching. For 10B21 steel Grade 8.8 bolts with M10 or larger and ML40Cr steel Grade 10.9 bolts with M27 or smaller, fast quenching oil should be used as the quenching and cooling medium; heating to 880°C or 890°C for quenching is recommended, with the tempering process remaining unchanged, resulting in a gradual improvement in mechanical properties.

4. Conclusion

The fatigue life of high-strength bolts has always been a major concern within the industry. Data indicates that the vast majority of bolt failures are caused by fatigue failure, which typically occurs with almost no warning signs. Fatigue crack initiation in some major accidents often occurs in areas where the heat-treated structure has not been fully quenched. Currently, the quality of heat treatment for high-strength bolts in China is problematic in some cases, with issues such as insufficient holding time during quenching, incomplete austenitization, and non-uniform microstructure. Therefore, it is essential to place great emphasis on metallographic examination of the quenched microstructure. ■