



Effects of Cu and Nb Addition on Microstructure and Mechanical Properties of Ultra-low Carbon Steel under Different Annealing Temperatures

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Citation:

Duc, N.T.; Son, N.C.; Dung, D.T.; Bui, A.H. Effects of Cu and Nb addition on microstructure and mechanical properties of ultra-low carbon steel under different annealing temperatures. *ASEAN J. Sci. Tech. Report.* **2026**, 29(10), e261425. <https://doi.org/10.55164/ajstr.v29i10.261425>

Article history:

Received: September 21, 2025

Revised: March 27, 2026

Accepted: April 11, 2026

Available online: September 20, 2026

Publisher's Note:

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Abstract: This paper investigated the beneficial effect of copper (Cu) and niobium (Nb) on grain size and mechanical properties of the ultra-low carbon (ULC) steel annealed at temperatures between 700 and 900 °C. The steel samples were prepared by melting input materials in an induction furnace, followed by hot forging, cold rolling into a thin plate, and annealing under an argon atmosphere. Mechanical testing and microstructural observations showed that the addition of Cu and Nb had a positive effect on the steel's properties. The grain sizes and the tensile strengths of the steel without Cu and Nb varied in 50-98 μm and 254-204 MPa, respectively, while those of the steel alloyed with Cu and Nb decreased to 40-83 μm and increased to 286-242 MPa at the same annealing temperatures. SEM analysis did not find Cu-enriched zones at the grain boundaries, consistent with the predicted Cu content in this steel. It can be concluded that Nb contributed to grain-size refinement and Cu hardened the ferrite matrix, leading to improved mechanical properties of ULC steel at the same annealing temperatures.

Keywords: Copper in steel; microalloyed by niobium; annealing process

1. Introduction

Current steelmaking processes can remove almost all harmful inclusions from steel products, except for tramp elements such as copper (Cu), which do not undergo any metallurgical reactions with others. As scrap is remelted multiple times, Cu accumulates in the steel. This leads to adverse effects on the mechanical properties of constructional steel, increased risk of hot tearing, and surface cracking as the Cu content rises [1, 2]. According to the iron (Fe) and Cu diagram, the solubility of Cu in Fe gradually decreases with lowering temperature. Because liquid steel solidifies slowly, saturated Cu tends to precipitate and concentrate near grain boundaries, deteriorating the mechanical properties of steel after hot rolling at 1050-1200 °C. Thus, it was acknowledged that the presence of Cu should be lower than 0.2 wt.% in steel; otherwise, it produces a particular characteristic, such as hot shortness when heated at elevated temperatures [2]. This is attributed to partial liquefaction in Cu-rich zones, as Cu melts at about 1083 °C. However, Cu in steel also has a positive effect, such as improving steel's corrosion resistance [3-6]. Zhou et al. noted that Cu precipitation enhanced the stability of retained austenite and the work-hardening rate, leading to increased elongation and strength after post-heat treatment [3]. In some cases, the strength of steel with a proper Cu content can

be increased because of ferrite strengthening by precipitation; however, this strengthening effect was thought to decrease with the increased C content [7-9]. Sellamuthu et al. found that Cu existed as a solid solution in as-cast low-carbon steel and precipitated during annealing; both strength and hardness increased with Cu addition [8]. According to Salvetti et al. [9], Cu precipitated out of a solid solution when the steel contained more than 0.5 wt.% Cu was reheated because the solubility of Cu was very low in the ferrite.

Ultra-low carbon (ULC) steel, containing less than 0.010 wt.% carbon, is widely used in the automotive and other industrial sectors. Due to its high ductility, this steel is used to produce cold-stamped components that require excellent surface quality and adequate strength. Manganese (Mn) and silicon (Si) are added to ULC steel to achieve a certain level of strength; however, they reduce ductility and toughness by dissolving into the steel matrix and distorting the crystalline network [10-12]. With these common alloying elements, a small amount of niobium (Nb) can be added to ULC steel to reduce grain size and harden the matrix phase through NbC formation, enhancing strength and ductility [13-15]. Unlike Cu, precipitated Nb compounds were found in annealed C steel, even when the Nb content was less than 0.03 wt.%; this precipitation was more pronounced in steels with higher C content [10]. Additionally, Nb can improve steel corrosion resistance. However, Nb content should be controlled at the micro-level (below 0.1 wt.%); otherwise, large grains will appear in the steel's matrix, causing stress concentration and deteriorating its mechanical properties [16]. With proper content, both Cu and Nb are expected to have a positive influence on steel's grain size and mechanical properties. For example, Kobayashi et al. proved that Nb and Cu existed independently in the steel's matrix; the effective hardening of Nb in the steel was 5 times larger than that of Cu [17]. The simultaneous alloying addition of Cu and Nb in steel is thought to lower the optimal heat-treatment temperature, balancing the steel's strength and toughness.

In practice, low Cu-containing scrap is prioritized for use in steel production to avoid harmful effects on the product. Although many studies have been conducted on ULC steel, the beneficial effects of Cu (a tramp element) and micro-alloying elements on this steel have not been well considered. Thus, this paper investigated the effect of Cu and Nb on the grain size and mechanical properties of ULC steel.

2. Materials and Methods

Two steel samples (M1 and M2) were prepared by melting the predetermined weight of pure metals, including iron (Fe), silicon (Si), manganese (Mn), copper (Cu), and niobium (Nb), in an induction furnace under an argon atmosphere. Amounts of the raw materials were calculated to achieve a normal ULC steel grade (0.01 wt.% C; 0.5 wt.% Mn; and 0.25 wt.% Si) with a content of Cu and Nb of about 0.1 wt.% for each element. Amounts of Cu and Nb were predetermined based on the above review of their impact (negative and positive) on the mechanical properties of steel. Chemical compositions of the round as-cast steel plates with 10 mm of thickness were analyzed by optical emission spectrometry (Spectromaxx LMF08), as given in Table 1. The main difference between these two steel samples was the Cu and Nb content: steel M2 had no Cu or Nb, while steel M1 contained 0.123 wt.% Cu and 0.108 wt.% Nb. A small deviation in the steel's chemical composition occurred; however, this was unavoidable due to loss during remelting.

Table 1. Compositions of the steel samples (wt.%).

Steel sample	C	Mn	Si	Cu	Nb	Fe
M1	0.010	0.490	0.244	0.123	0.108	Balance
M2	0.010	0.491	0.254	-	-	Balance

In practice, ULC steel slab is hot-rolled through several passes to obtain a desired thickness, at which its temperatures are in the range of 700 - 900 °C, and then slowly cooled to room temperature. This study used initial hot forging instead of hot rolling because a hot-rolling machine was unavailable. The as-cast steels were hot-forged to a thickness of 3 mm at 900 °C, cooled to room temperature, then cold-rolled into a 2 mm sheet through 2 passes, and annealed at various temperatures from 700 to 900 °C under an argon atmosphere. The annealing condition was fixed at 30 minutes of holding time, with slow cooling in the furnace. The specimens for the tensile test (MTS E45.105) and microstructural observation were cut along the rolling direction, as illustrated in Figure 1. Some steel specimens were ground, polished, 2% nital-etched, and observed using an

optical microscope (Olympus, GX53). Grain size was measured by the linear intercept approach, in which a line was superimposed over the optical microstructure. The true line length was divided by the number of grains intercepted by the line. This gave the average line length within the intercepted grains. The average grain size of the steel was obtained from five measurements. The micro-hardness was measured using Wolpert Wilson Instruments 401/402 MVD with a load of 1 kgf for 5 seconds. Scanning electron microscopy (SEM, JEOL 7600F) was used to observe the precipitation in the annealed steel.

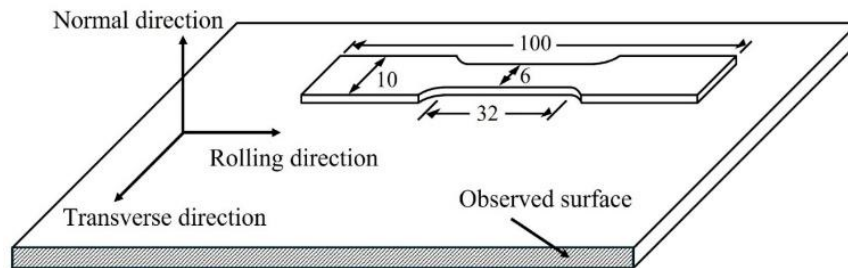
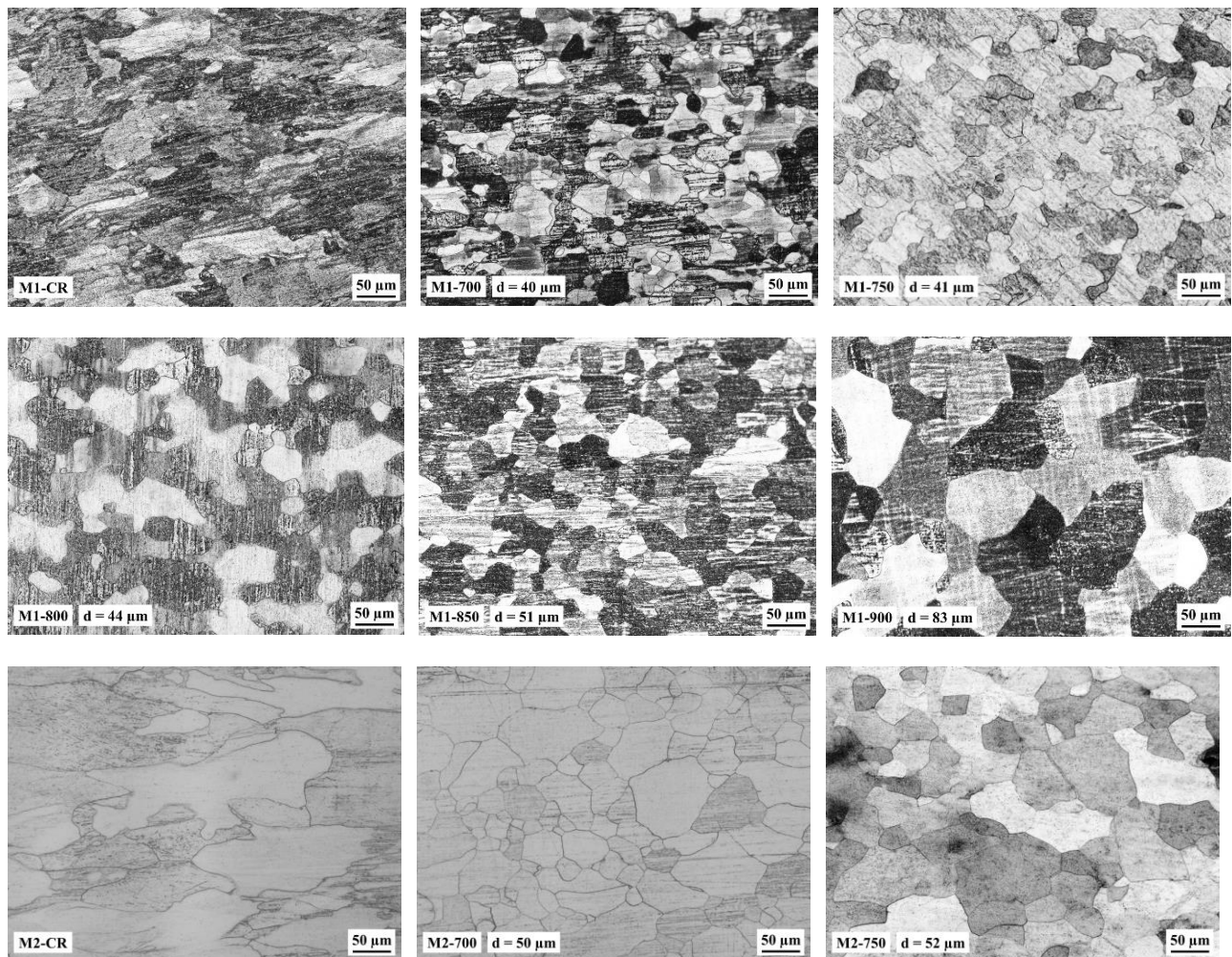


Figure 1. Cutting position of specimens for tensile test and microstructural observation.

3. Results and Discussion

3.1 Microstructure



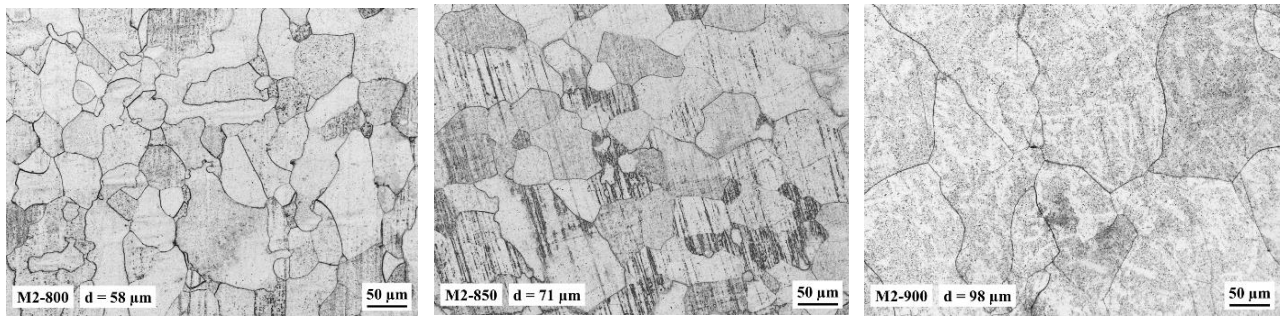


Figure 2. Microstructure of the cold-rolled and annealed steels ($\times 200$).

Microstructure is essential to the mechanical properties of steel. Figure 2 shows the optical images of the cold-rolled and annealed steels, in which ferrite grains were seen for both ULC steels (M1 and M2) with a C content of 0.01 wt.%. For the cold-rolled steels, the ferritic grains tended to lengthen along the rolling direction. However, a transformation from the microstructure of the long ferrite grains to a homogeneous microstructure occurs after annealing the steels at the given temperatures of 700–900 °C. Reducing cold rolling decreased the recrystallization temperature of steel and shortened the time required to complete the process [18]. Although the recrystallization temperature was considered to depend on the content of alloying elements [17], the temperatures of steel M1 and M2 were estimated not to be significantly different because steel M1 contained a small amount of Cu and Nb. Recrystallization was thought to occur completely at these annealing temperatures. In steel M2 annealed at 700 °C, the grain sizes were different. At higher annealing temperatures, the homogeneously coarsened microstructure was observed.

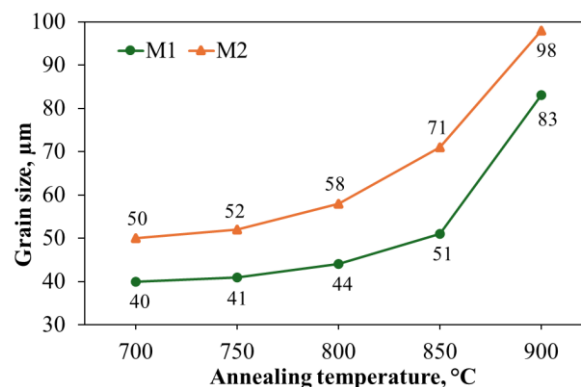


Figure 3. Grain size of steel M1 and M2 versus the annealing temperature.

In steel M1 with Cu and Nb, the microstructures were more homogeneous, with a finer grain size at all annealing temperatures. As plotted in Figure 3, the grain size annealed at 700 °C was 40 μm for steel M1 compared to 50 μm for steel M2 with no Cu and Nb. Notably, the grain size of steel M1 annealed at 850 °C was almost the same as that of steel M2 annealed at 700 °C. Consistent with other publications [13–15], this result was attributed to the grain-refining effect of Nb in the steel, due to grain-boundary pinning at the annealing temperatures. Thus, the mechanical properties of steel M1 were expected to improve. At higher annealing temperatures (800–900 °C), the grain size of both steel M1 and M2 tended to increase. However, the grain size of steel M1 with Nb-Cu was always smaller than that of steel M2 without these elements at the same annealing temperatures. In particular, the grain size of both steel samples increased significantly at 850 and 900 °C due to strong recrystallization. Once the grain boundaries became more flexible at these high temperatures, it was easy for smaller grains to merge and form abnormally large ones, even in the Nb-containing steel M1 annealed at 900 °C.

Additionally, SEM observations revealed no Cu precipitation at the grain boundaries or inside the ferrite grains for all steel samples, even in the steel M1 annealed at 900 °C, as shown in Figure 4. This can be

explained by the fact that the Cu content in steel M1 was not high enough for Cu to precipitate out from the ferrite [9]. It can be concluded that the presence of Nb has hindered the coarsening phenomenon, and dissolved Cu strengthened the ferrite grains of the annealed steel M1, which is expected to cause good mechanical properties in the steel.

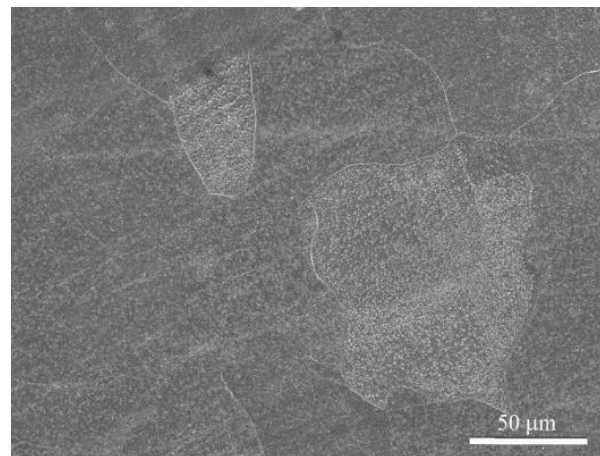


Figure 4. SEM image of the steel M1 annealed at 900 °C.

3.2 Mechanical properties

It is acknowledged that the distortion of the crystalline structure by cold deformation also contributed to hindering dislocation movement, thereby affecting the steel's deformation behavior. Once the annealing process effectively removes work hardening, dislocation density decreases significantly, leading to decreased strength and increased plasticity. Figures 5 and 6 show the stress-strain curves and the change in the strength of the steel processed under different conditions. The relatively large cold reduction (33%) increased the strength of the steel while reducing its ductility, consistent with published results [8, 19]. This can be explained by cold deformation increasing dislocation density and steel defects, resulting in increased residual energy within the steel. The change in the steel's mechanical properties with increasing annealing temperature can be predicted from microstructural images. For example, changes in grain size and homogeneity affect mechanical properties. Typically, steel strengthening mechanisms include grain refinement and precipitation of a hard phase. To explain the strength of steels M1 and M2 in this study, the large grain size decreased strength, while inhomogeneity reduced the elongation of the present steel. Besides the pinning grain boundary effect of Nb, the presence of Cu in the steel also contributed to the hardening of the steel's matrix phase, resulting in higher hardness of the steel M1 compared to the steel M2 under all experimental conditions.

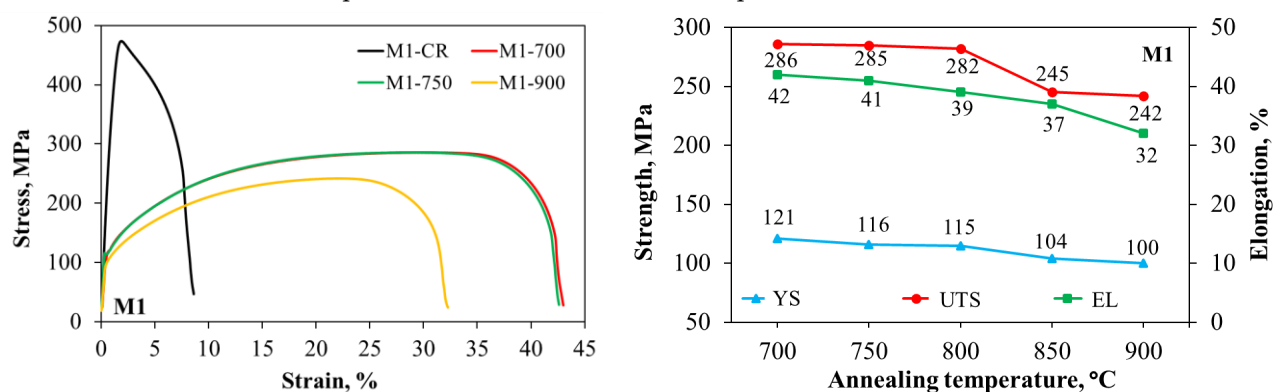


Figure 5. Typical stress-strain curves and the change in the strength of the annealed steel M1.

As shown in the calculated grain size (Figure 3) and tensile results (Figures 5 and 6), a higher annealing temperature yielded a larger grain size and decreased the strength of the steels. In general, a heterogeneous microstructure affected the mechanical properties of the steel; for example, coarsened grain zones deformed more easily than finer grain zones because fewer grain boundaries hindered dislocation movement. This was

attributed to concentrated partial stress, which resulted in earlier failure in the steel and reduced total elongation. The acknowledged trend for steel M1 and M2 was higher plasticity and lower strength at higher annealing temperatures. Both steels showed good ductility; the highest elongation was achieved at 700 °C for steel M1 (42%) and at 900 °C for steel M2 (41%). This indicated that the mostly ferritic soft matrix ensured sufficient plasticity for both steels at different annealing temperatures. For steel M1 annealed at 700 °C, a homogeneous microstructure with the smallest grain size was attributed to the highest strength and elongation. With a low content of Cu and Nb, steel M1 still maintained a balance between strengthening and softening effects and had good mechanical properties. However, this Nb content could not keep this balance at an annealing temperature of 900 °C, resulting in a drastic decrease in hardness. For steel M2 annealed at 900 °C, the homogeneously coarsened microstructure caused better ductility and a slight decrease in strength.

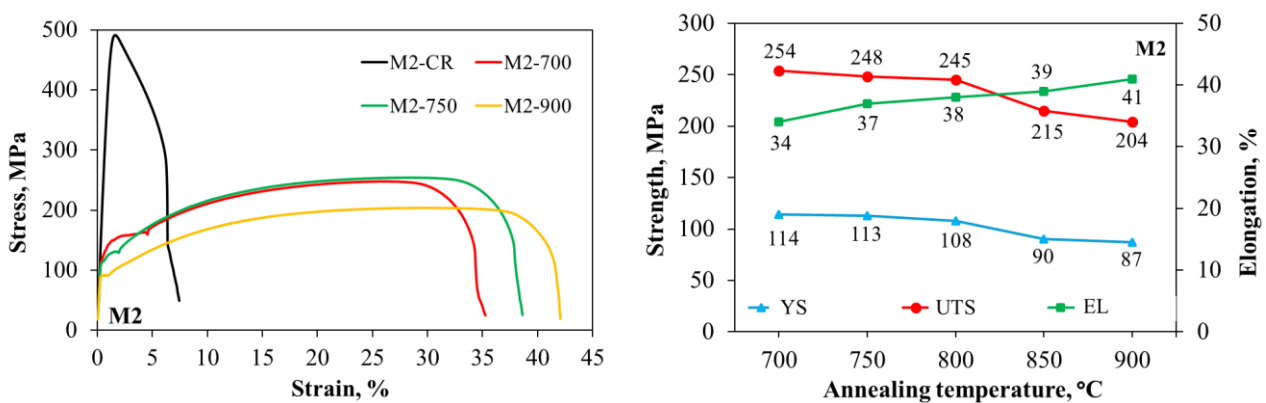


Figure 6. Typical stress-strain curves and the change in the strength of the annealed steel M2.

3.3 Hardness

The hardness and strength of steel are influenced by alloyed elements, grain size, and dislocation density [20-23]. As shown in Figure 7, the hardness of both steels decreased significantly after being annealed at 700-900 °C. Hardness was highest in the cold-rolled steel and decreased in the annealed steel. This reduction was attributed to a decrease in dislocation density and the release of stored energy accumulated in the cold-rolling process. When the annealing temperature increased from 700 to 900 °C, the hardness of both steel M1 and M2 exhibited a gradual decrease, and steel M1 had higher hardness than that of steel M2. Theoretically, the variation in strength and hardness with annealing temperature can be attributed to ferrite softening or strengthening and to matrix homogeneity. A decrease in hardness with the annealing temperature was found in both steel M1 and M2, caused by the coarsened grains and heterogeneity of the steel's matrix (as seen in Figure 2). Grain coarsening could explain the decrease in hardness as the annealing temperature increased. However, a higher hardness of cold-rolled steel M2 (without Cu and Nb), compared to that of the cold-rolled steel M1 (with Cu and Nb), was obtained. This phenomenon can be explained, as Syarif et al. confirmed, by the Cu strengthening effect on the ferrite matrix, which occurred only at low dislocation densities [24]. Thus, the presence of Cu in the cold-rolled steel M1 harmed its hardness.

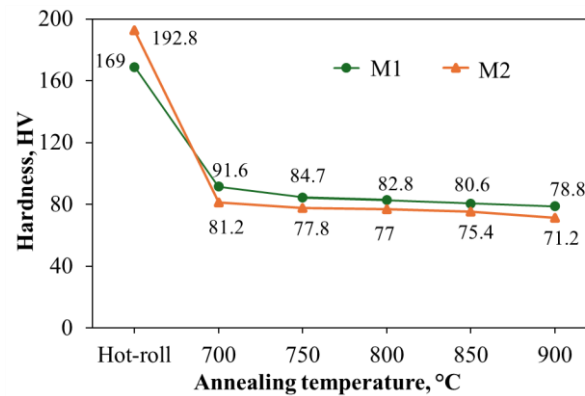


Figure 7. Hardness of steel M1 and M2 versus the annealing temperature.

4. Conclusions

This study investigated the beneficial effects of adding 0.123 wt.% Cu and 0.108 wt.% Nb on the grain size and mechanical properties of ULC steel containing 0.010 wt.% C. Adding Cu and Nb slightly reduced the strength and hardness of the cold-rolled ULC steel but significantly improved the strength of the annealed steel. The grain size of both steel samples increased with higher annealing temperatures; however, the presence of Cu and Nb effectively refined the grain size. Namely, the grain size ranged from 40–83 μm for steel M1 with both Cu and Nb, compared to 50–98 μm for steel M2 without these elements, corresponding to annealing temperatures from 700 to 900 °C. This refinement contributed to high strength and hardness in steel M1 annealed at 700–800 °C, but to lower values at 850–900 °C. This finding is relevant to the practical production of ULC hot-rolled coil (HRC), which is typically stored and cooled slowly within this temperature range to achieve the desired mechanical properties. At an annealing temperature of 700 °C, the steel M1 exhibited the highest UTS of 286 MPa and elongation of 42 %, while steel M2 achieved a maximum UTS of 254 MPa and the lowest elongation of 34 %. These results will help steelmakers apply appropriate processing for different applications of ULC steel.

5. Acknowledgments

Author Contributions: Conceptualization, Anh-Hoa Bui; methodology, Nguyen Cao Son and Anh-Hoa Bui; validation, Nguyen Trung Duc and Do Thanh Dung; investigation, Nguyen Trung Duc and Do Thanh Dung; writing—original draft preparation, Nguyen Trung Duc; writing—review and editing, Nguyen Cao Son; supervision, Anh-Hoa Bui. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Conflicts of Interest: The authors declare no conflict of interest.

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