Low-Carbon, Cu-Precipitation-Strengthened Steel

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INTRODUCTION

Our national infrastructure and other structural applications can greatly benefit from steels with increased strength but without sacrificing ductility, toughness and weldability. Most high-strength steels are martensitic. The strength of martensitic steels increases with carbon content. High carbon content, however, leads to poor weldability due to the formation of a brittle heat-affected zone adjacent to the weld. One can overcome this problem by using steels with low carbon content and enhancing the strength by precipitates. This was the basis for the development of HSLA-80 and HSLA-100 Cu-precipitation-strengthened steels [1-6] now used in Naval applications, mining and dredging equipment, heavy duty truck frames and are beginning to be used in bridge applications.

It has been known since the 1930's that the addition of Cu to steels leads to precipitation strengthening due to the formation of small (few nanometers) Cu-containing clusters. Such nano-sized Cu alloy clusters or precipitates markedly increase the strength of the base metal. In addition to their effects on strength, these nano-sized coherent-co-planar precipitates improve the mobility of screw dislocations at low temperatures, resulting in lower ductile-to-brittle transition temperatures and higher fracture energies[7]. Investigations of copper precipitation in steel have been carried out at Northwestern University in as-rolled and air-cooled and aged conditions. This paper is focused on the effects of composition, soaking temperature before hot-rolling and heat-treatment on mechanical and fracture properties of copper-precipitation-strengthened steels.

EXPERIMENTAL PROCEDURE

Our investigations began with a number of laboratory heats of 45 to 135-kg size produced at Inland Steel Research Laboratory (now ArcelorMittal Global R&D) and US Steel Research Center. Subsequently, the steel was produced commercially three times; twice by Oregon Steel Mills (OSM) at Portland, OR and once by International Steel Group (ISG) (now ArcelorMittal) steel plant at Coatesville, PA. One of the OSM production heats was used for seismic retrofitting of a bridge in Southern Illinois and five heats from ISG were used for the construction of a bridge in Northern Illinois. All commercially produced steels were hot-rolled, and then air-cooled. The thickness of the plates varied from 3 to 52 mm. In addition, one ingot from OSM (heat OSM-2) was hot-rolled at U.S. Steel Plate Products, Gary, IN to study the effect of water quenching and aging on steel properties.

Standard tensile specimens with gage section of 32 or 51 mm were used to measure the yield, ultimate tensile strength and elongation to failure. Standard full-sized Charpy specimens were used to measure absorbed fracture energy. The Charpy specimens were cooled own in methanol or acetone with dry ice when tested at below room temperature. Steel samples for optical microscopy studies were polished down to 1 micron and then etched in a 5% nital solution.

RESULTS AND DISCUSSION

The results of the steel development are divided into two parts. The first part is a discussion of the laboratory steel production, and the second part deals with commercial steel production.

Laboratory Heats

The primary aims for these investigations were to study the effects of steel composition, soaking temperature before hot-rolling, and steel thickness on the mechanical and fracture properties of the steel. A large number of laboratory heats were produced, but only a few are discussed in this paper. The laboratory steel heats marked as NUCu-I-X were produced at Inland Steel (now ArcelorMittal); the laboratory steel heats marked as NUCu-USS-Y were produced by USS. The compositions of the selected steels are given in Table I. To improve welding properties, carbon concentration was kept below 0.07% in all the heats. The steels contained copper in the 1.16 to 1.49% range. Nickel was added to the steel in the 0.4 to 0.97% range to prevent hot-shortness during hot rolling. Niobium was added in the 0.03 to 0.08% range for grain refinement. Titanium was intentionally added to NUCu-USS-4 to NUCu-USS-6 steels to study the effect of Ti on ductile-to-brittle transition in steels.

Table I. Compositions of laboratory steels (wt. %)

	C	Cu	Ni	Mn	Si	Nb	Ti
NUCu-I-1	0.02	1.16	0.44	0.54	0.80	0.062	NM
NUCu-I-7	0.06	1.32	0.84	0.49	0.50	0.081	NM
NUCu-USS-2	0.06	1.37	0.97	0.82	0.40	0.036	0.02
NUCu-USS-4	0.05	1.30	0.90	0.50	0.39	0.060	0.03
NUCu-USS-5	0.06	1.29	0.90	0.50	0.39	0.060	0.07
NUCu-USS-6	0.06	1.30	0.90	0.50	0.39	0.060	0.10

NM – not measured

NUCu-I-1 steel (3.1 mm thick plate) was used for the preliminary evaluation. On air-cooling from hot rolling, yield strength of 559 MPa and ultimate tensile strength of 704 MPa and total elongation of 25% (Table II) were obtained. The yield strength increases to 700 MPa after aging for 30 mins at 500°C. Formability was investigated by the dome height to failure test. With a 45 mm diameter punch, the steel did not fail up to 20 mm, the limit of the apparatus, showing the steel to have very good cold-forming properties.

Copper precipitation hardening is a significant contributor to strength of this steel and as expected is influenced by aging temperature and time. For example, the influence of aging on hardness at two temperatures for NUCu-I-7 steel that was austenitized at 900°C and air-cooled is shown in Figure 1. The steel ages much faster and reaches the peak hardness in less than 20 minutes of aging at 600°C but it takes 480 minutes of aging at 500°C.

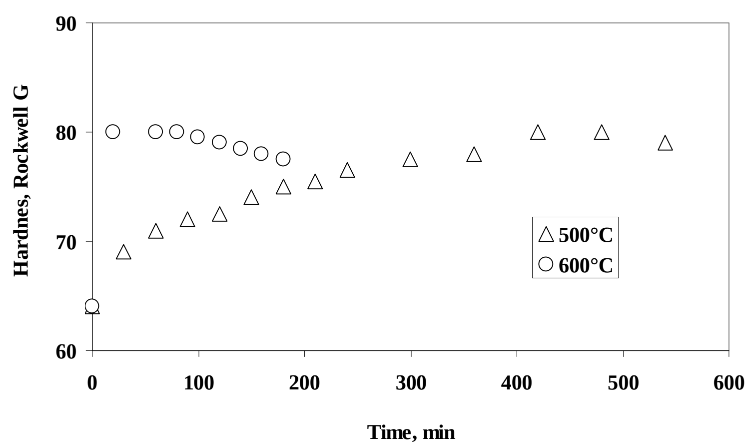

Figure 1. Age hardening of NUCu-I-7 steel at two temperatures after austenization at 900°C and air-cooling[8]

The yield strength of the as-rolled and air-cooled laboratory steels varied from 480 to 559 MPa, the UTS varied from 545 to 616 MPa and elongation to failure varied from 25 to 31% (Table II). These variations most likely resulted from differences in hot rolling conditions, implying the need to closely control chemistry and processing conditions to achieve desired set of properties.

Table II. Tensile properties of steels in as-received condition

Steel	Thickness, mm	Soaking Temperature, ºC	0.2% Offset Yield, MPa	UTS, MPa	Elongation to Failure, %
NUCu I-1	3.2	NC	559	704	25
NUCu-I-7	12.7	NC	525	616	30
NUCu-USS-2	50.8	1150	490	552	31
NUCu-USS-2	50.8	1230	511	593	28
NUCu-USS-4	12.7	1150	480	545	30
NUCu-USS-5	12.7	1150	507	590	28
NUCu-USS-6	12.7	1150	493	583	28

NC --- Not controlled or not measured

Titanium is known to be an excellent scavenger for carbon interstitials, forming TiC particles. Removal of carbon interstitials is expected to minimize the pinning of dislocations, thereby improving their mobility and increasing the low-temperature fracture toughness. The effect of Ti on mechanical and fracture properties of steel was investigated in laboratory heats (NUCu-USS4- NUCu-USS6) where Ti content varied from 0.03 to 0.10 wt.%. The effect of increasing the Ti from 0.03 to 0.10 wt. % on the microstructure is shown in Figure 2. Ti appears to have little effect on the ferrite grain size. As noted earlier, titanium reacts with carbon to form TiC and practically all pearlite disappeared when 0.1 Ti was added to steel. It is well known that pearlite reduces the fracture toughness of steels.

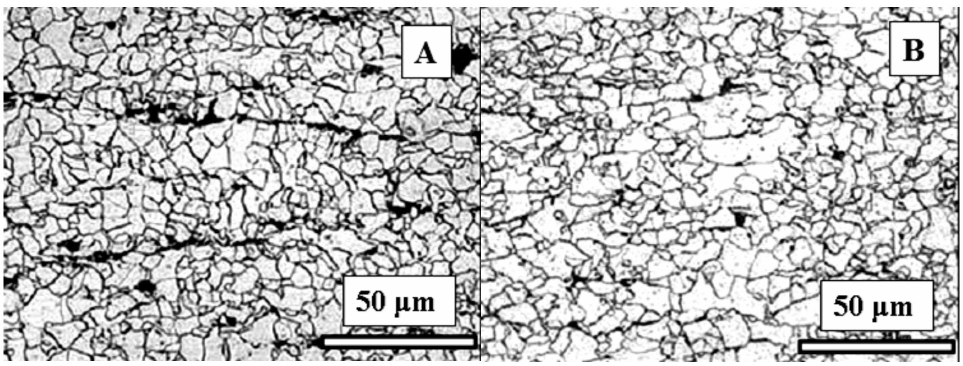

Figure 2. Optical micrographs: (A) NUCu-USS4 steel with 0.03 wt. % Ti and (B) NUCu-USS6 steel with 0.10 wt. % Ti

The Charpy absorbed fracture energies for these steels are shown in Figure 3. The arrows in Figure 3 represent specimens that did not break in the Charpy apparatus, indicating that the true fracture energy for these specimens is greater than 358 J, the limit of the Charpy machine. These specimens bent and were only partially fractured with ductile fracture surfaces. At 24ºC, none of the steels failed. At -22ºC, Charpy specimens made of steels containing 0.07 and 0.10% Ti did not fracture. At -40ºC, specimens made of steel containing 0.10% Ti did not fracture. The unusually high fracture energy at the higher test temperatures must have resulted from the fracture stress being significantly higher than the yield strength. As shown in Figure 3, the >358 J level shifts to a lower temperature with increased Ti content.

The laboratory steels were obtained by vacuum induction melting and were then hot-rolled and air-cooled. The soaking temperature before hot rolling was not controlled when the Inland Steel heats were processed. For later heats, soaking temperature was in general kept below 1150ºC. However, for the USS2 heat, a 1230ºC soaking temperature was used. Analysis of data from Table II indicates that higher soaking temperature before hot-rolling (when the composition and thickness of the steel are kept the same) in general leads to higher strength. For example, NUCu-USS-2 steel pre-heated to 1230ºC had yield strength of 511 MPa and UTS of 593 MPa versus 490 MPa and 562 MPa for steel pre-heated to 1150ºC before hot-rolling. However, the Charpy absorbed fracture energy after a higher soaking temperature was worse than that for steels with lower soaking temperature: the Charpy energy at room temperature is only 35 J. As shown in the following section, this is due to formation of a coarse and brittle Widmanstatten ferrite at higher soaking temperatures. Interestingly, the excellent impact toughness property can be restored by a low-temperature normalizing heat treatment. For example, steel samples that were originally soaked at 1250ºC for 30 minutes and air-cooled, have been reheated (normalized) for 30 minutes at different temperatures from 500 to 1100ºC range. Figures 4a and 4b show the effects of this treatment on the strength and Charpy absorbed impact fracture energy of the steel. Normalizing heat treatment above 700ºC leads to reduced yield and ultimate tensile strengths, and simultaneous increase in Charpy energies. Indeed, normalizing treatments between 950 and 1050ºC resulted in steels that did not fracture in the Charpy apparatus.

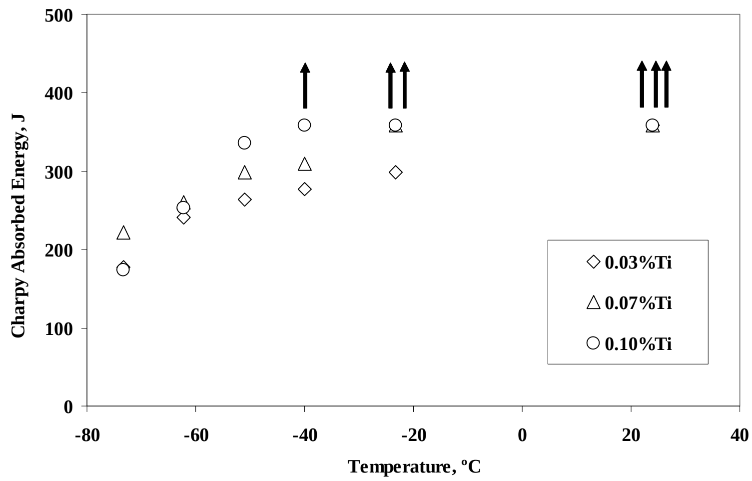

Figure 3. Variation of Charpy absorbed fracture energy as a function of temperature for three NUCu-USS steels modified by Ti. Arrow indicates that specimen did not fracture.

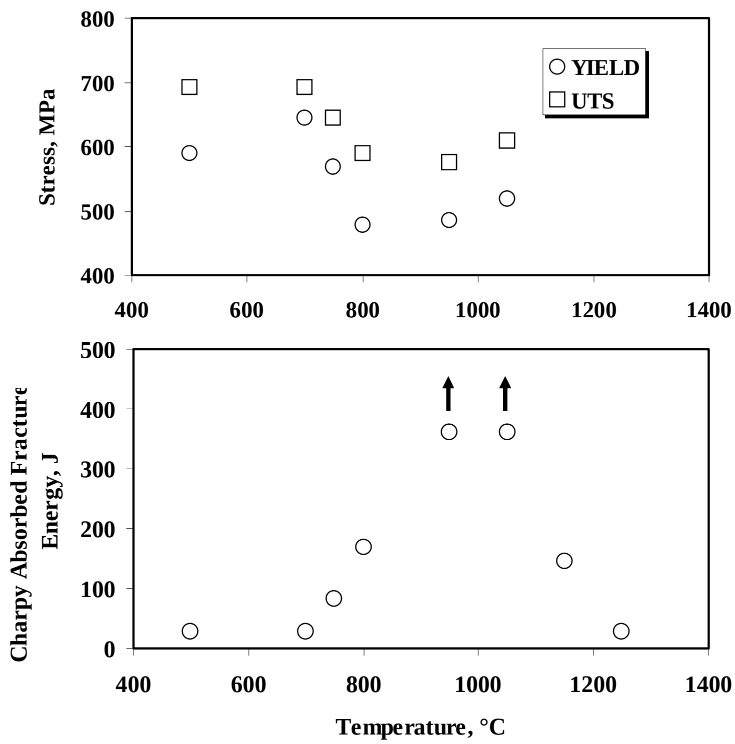

Figure 4. Effect of re-heating (normalizing) temperature (30-min.) on room-temperature strength and Charpy absorbed impact fracture energy. Specimens were initially heated 30 minutes at 1250^{o}C and air-cooled. After reheating, specimens were air-cooled. Arrow indicates that specimen did not fracture.

Commercial heats

The compositions of the commercial heats investigated are shown in Table III. The compositions of OSM-1 and OSM-2 heats differed in the amounts of C, Cu, Mn and Nb. The compositions of all five ISG heats were in a very narrow range.

Table IV shows the mechanical properties of steel plates produced at OSM. Five plates ranging in thickness from 12.7 to 50.8-mm-thick were hot-rolled from the first heat. Two plates, 19.2 and 25.4-mm-thick, were rolled from the second heat. It is obvious that the steel plates rolled from the first heat are stronger than the plates rolled from the second heat. This is most likely due to the higher concentration of C, Cu and Nb in the first heat. Figure 5 shows the tensile properties of 35 steel plates produced at ISG. The yield strength varied from 490 to 670 MPa and ultimate tensile strength varied from 570 to 720MPa. Although there is some scatter in the data, a general trend can be noted: the thicker the steel plate, the lower the yield and ultimate tensile strengths. Since the steel is produced by routine air-cooling after hot rolling, the plate thickness affects the cooling rate of a plate, i.e., thicker plate cools down slower than thinner plate. Therefore, the plate thickness indirectly affects the size of copper and niobium carbide precipitates and thus the mechanical properties of the steel. Figure 6 shows the Charpy absorbed fracture energy at -23ºC for plates produced at ISG. The Charpy absorbed fracture energies for most of the plates were over 150 J at -23ºC, significantly exceeding the 47 J required by ASTM A709 Standard for fracture-critical bridge members.

Table III. Compositions of commercial steels (wt. %)

	C	Cu	Ni	Mn	Si	Nb	Ti
OSM-1	0.03	1.49	0.84	0.49	0.40	0.062	0.03
OSM-2	0.06	1.37	0.80	0.78	0.38	0.038	0.03
ISG*	0.06-0.07	1.33-1.37	0.69-0.70	0.69-0.70	0.41-0.44	0.035-0.037	0.02

*Composition ranges for five 100-ton heats are given.

Table IV. Tensile properties of OSM steels

Steel	Thickness, mm	Soaking Temperature, ºC	0.2% Offset Yield, MPa	UTS, MPa	Elongation to Failure, %
OSM-1	12.7	1150	611	638	28
OSM-1	16.0	1150	587	673	26
OSM-1	19.1	NC*	597	697	26
OSM-1	25.4	1150	549	611	29
OSM-1	50.8	1150	490	552	29
OSM-2	19.1	1150	504	566	32
OSM-2	25.4	1150	469	552	32

*NC --- Not controlled

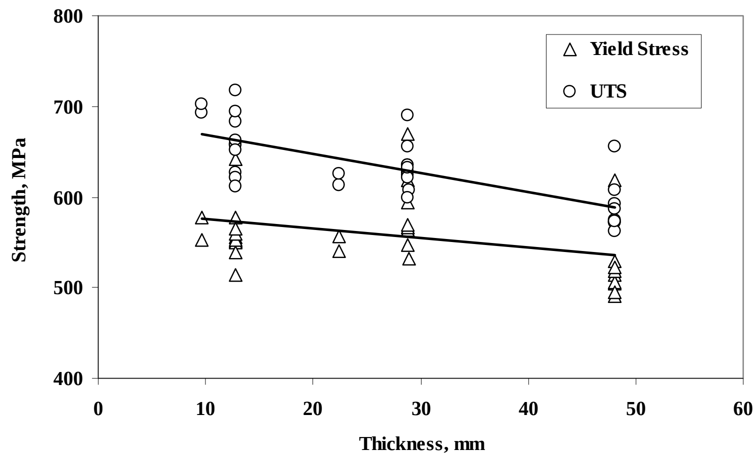

Figure 5. Relationship between yield strength and UTS and thickness of the plates produced by ISG

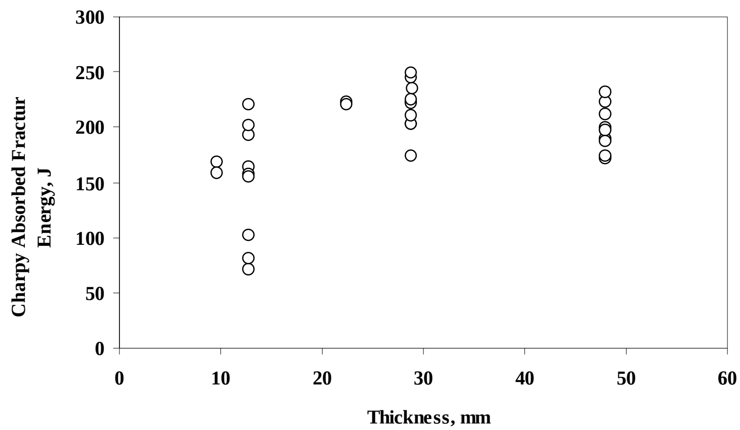

Figure 6. Charpy absorbed fracture energy at -23ºC for plates produced by ISG

As mentioned before, experiments with laboratory heats showed that steels produced with soaking temperatures not exceeding 1150ºC had very good Charpy absorbed fracture energy values. The same is observed for commercial heats. For example, Figure 7 shows the variation of Charpy absorbed fracture energy with temperature for two OSM-1 plates. The 12.7-mm-thick plate was soaked at 1150ºC before rolling and 15.9-mm-thick plate was overheated during rolling due to steel mill's operational difficulties. The Charpy absorbed fracture energy is very good for the 12.7-mm-thick plate, exceeding 100 J even at -63ºC. On the other hand, the fracture energy for the 15.9-mm-thick plate (which was overheated) is lower at all temperatures, especially at -40 and -63ºC. This plate was then subjected to normalizing treatment at 950ºC for 30 minutes and air-cooled. Figure 8 shows the effect of normalizing on the Charpy energy for this plate. Note the marked improvement in impact toughness: all Charpy specimen tested down to -40ºC did not fracture.

This copper-precipitation strengthened steel hot rolled from lower than 1150°C soaking temperature is ferritic with a small amount of pearlite. When the steel is overheated prior to hot-rolling, Widmanstatten ferrite (which is brittle) is formed (Figure 9A). Normalizing the plate for 30 min at 950ºC leads to recrystallized equiaxed ferrite grains (Figure 9B) and to excellent Charpy absorbed energy as shown in Figure 8.

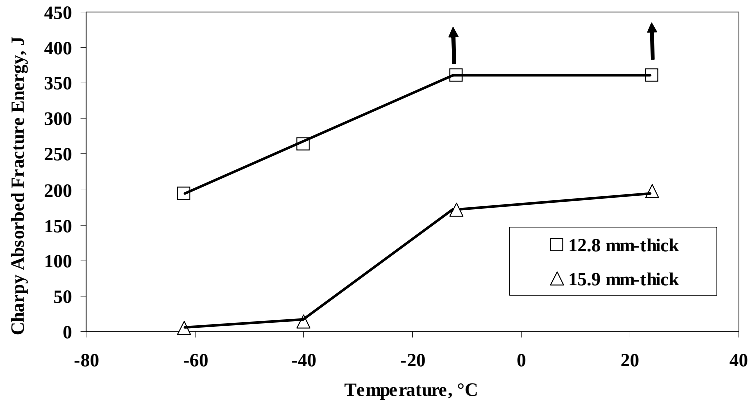

Figure 7. Effect of testing temperature on Charpy absorbed fracture energy of two plates produced at OSM. Arrow indicates that specimen did not fracture

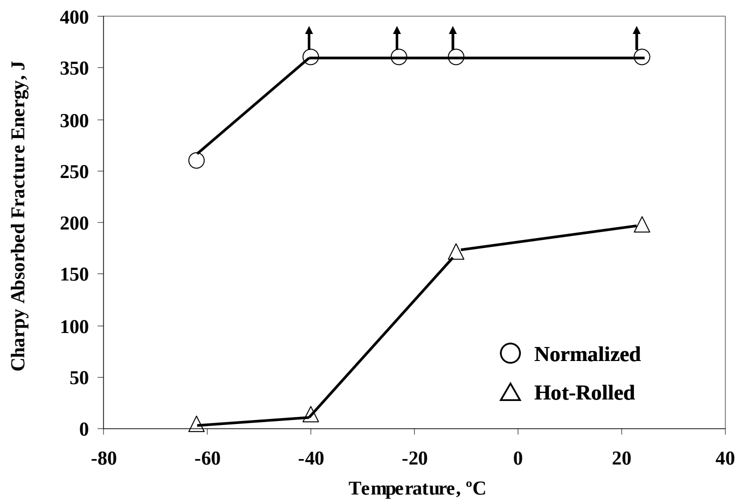

Figure 8. Effect of normalizing (30 minutes at 950ºC) on Charpy absorbed fracture energy of 15.9-mm-thick OSM-1 plate. Arrow indicates that specimen did not fracture

Effect of Aging on Mechanical and Fracture Properties

The steel is produced by hot-rolling and subsequent air cooling. The final mechanical properties of the steel plates are determined by the size, number density and distribution of the copper-rich precipitates formed during this process. Properties of the steel can be further adjusted by aging. The ingot of OSM-2 heat was rolled into plates at US Steel Company. The steel was austenitized at 900°C and water-quenched. The time at temperature for austenitizing was 40 minutes per 25.4-mm thickness. The steel was aged for 1 hour.

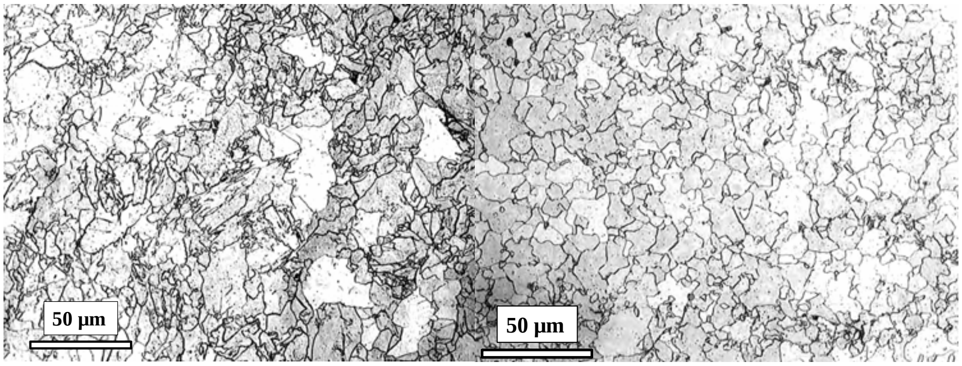

Figure 9. Microstructure of 15.9-mm-thick OSM-1 plate: A – as-hot-rolled, B – normalized 30 minutes at 950ºC

Aging of as-rolled 19.1-mm- thick plate at 525°C increased the strength of the steel by about 35-45 MPa (Table V) with a slight reduction in the Charpy absorbed impact energy (Figure 10). The quenching and aging increases the strength by a larger margin, but as expected the absorbed fracture energy is lower in this case, i.e. higher strength results in lower ductility and fracture toughness.

Table V. Mechanical properties of 19.1 mm-thick plate

Plate Condition	0.2% Offset Yield, MPa	UTS, MPa	Elongation to Failure, %
As-Rolled	503	567	32
As-Rolled & Aged at 524°C	539	610	33
Quenched& Aged at 524°C	712	780	26

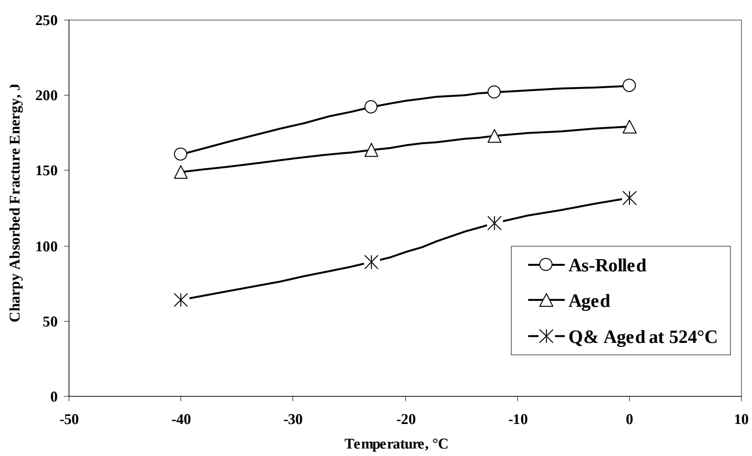

Figure 10. Charpy absorbed fracture energy of as-rolled and as-rolled and aged at 524°C

As is obvious, the microstructure of the steel changes as a function of heat-treatment (Figure 11). In the as-rolled condition, the steel has an equiaxed ferritic microstructure with small regions of pearlite, which forms bands parallel to the surface of the plates. Pearlite is not present in the quenched and aged steel. While the average grain size is approximately 12-15 µm in hot-rolled steel, the grains in quenched steel are significantly smaller, on the order of few microns. Reduction in grain size contributes to the strength of the steel in addition to strengthening from copper precipitate due to aging. Copper precipitates could not be observed in the optical microscope. Separate measurements by 3D atom probe showed that they are approximately 3 nm in diameter after these treatments[7].

SUMMARY

This research has shown that copper-precipitation-strengthened steels with excellent combination of strength and fracture toughness can be produced by cooling from hot-rolling. Strength of the steel can be further increased by aging or by quenching and aging without significant reduction in fracture toughness.

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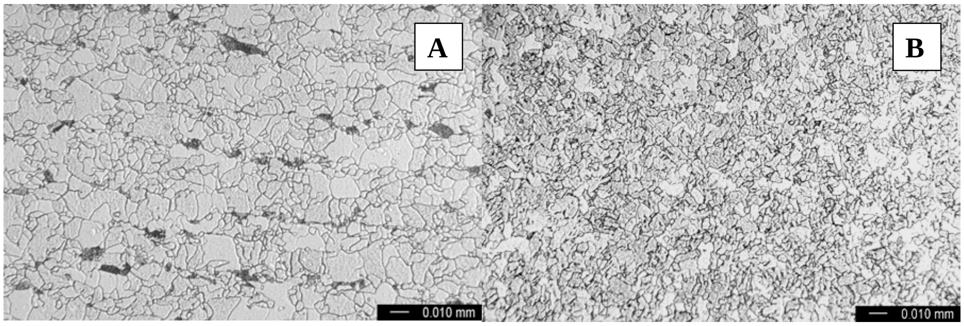

Figure 11. Microstructure of steel plate rolled from OSM-1 ingot at US Steel Company:
A – as-hot-rolled and air-cooled, B – water-quenched from 950°C and aged at 524°C

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