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DEVELOPMENT AND PRODUCTION OF ULTRA-HIGH-STRENGTH STRUCTURAL STEEL S1100QL

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Abstract: *High-strength structural steels present the backbone for the design of the modern constructions, as they are incorporated in different components in transportation, mining, infrastructure and lifting industries. Various standards prescribe the specific requirements for mechanical properties, testing as well as limitations for chemical composition and production. Nowadays, as there are progressively stricter demands for lighter, stronger, and safer structural components, non-standardized steel grades are required. Prime example is quenched and tempered ultra-high-strength structural steel S1100QL with yield strength exceeding 1100 MPa. The main challenge when designing and producing such demanding steel grade is not the yield strength itself, but a combination of high-strength and toughness with good workshop properties like weldability and bendability. Only a specific combination of different metallurgical mechanisms, such as grain refinement, solid solution strengthening, work hardening and precipitation strengthening, can provide the desired properties. The paper presents crucial steps when developing S1100QL steel grade. Different microalloying additions in combination with lean chemical composition were first tested on the laboratory scale to set up optimal composition for the industrial production. Main challenges during the industrial production of quarto plates were addressed through carefully controlled steelmaking with steel scrap recycling, secondary metallurgy, continuous casting, thermo-mechanically controlled hot rolling, and heat treatment process. Final mechanical properties in quenched and tempered condition were tested and verified.*

Keywords: *S1100QL, ultra-high-strength steel, microalloying, mechanical properties, quarto plates*

1. INTRODUCTION

Current and future trends for components made of high-strength structural steels follow the path of increasing material payload, durability and reducing the weight. Prime example is lifting industry, where the combination of high load-bearing capacity and low weight is crucial for safe and efficient operation of mobile, loader and tower cranes. Booms for such components are exposed to stresses which can exceed yield strength ($R_{p0,2}$) of common structural steels, resulting in catastrophic failure. This issue can be addressed by increasing either the thickness or the strength of the boom.

Nowadays, booms are made of high-strength structural steels in quenched and tempered (QT) condition, specified in standard EN 10025-6 [1], which entitles grades with $R_{p0,2}$ from 460 to 960 MPa. Generally, higher strength levels are used, namely 890 or 960 MPa, however, as there is ever growing need for lighter and stronger booms, non-standardized steel grades are being developed and used. Example of such steel grade is S1100QL, with $R_{p0,2}$ exceeding 1100 MPa. The term ultra-high-strength is not standardized and is used only to differentiate from high-strength steel grades specified in EN 10025-6. The biggest challenge when designing such steel grade is not high-strength alone, but the combination with toughness and workshop properties like weldability and bendability.

To obtain the desired properties, various metallurgical mechanisms are incorporated, like grain refinement, work hardening, solid-solution strengthening and precipitation strengthening [2]. All mentioned mechanisms depend on the chemical composition and production technology. Chemical composition should be as lean as possible to assure good weldability and bendability, however sufficient hardenability and grain size control shall be obtained. One way to obtain sufficient grain size control is with the use of the microalloying additions, especially Nb and Ti, which pin the prior austenite grain boundaries with nano-sized precipitates during reheating and hot rolling. Ti is also used for B protection, which is intentionally added for better hardenability, along with Cr, Mn and Mo. Ni addition is used to increase the low-temperature toughness, as it improves the mobility of dislocations within the iron lattice.

The aim of this paper is to present the concepts and challenges of designing ultra-high-strength steel grade S1100QL from laboratory to industry scale. Different alloying and microalloying additions were used and evaluated to set up the optimal chemical composition for industrial trials, where final mechanical properties in heat-treated condition were determined. Holding at various elevated temperatures for 1 h was done to determine maximum safe temperature, without degradation of mechanical properties.

2. EXPERIMENTAL

2.1 Laboratory scale

Production process

Three laboratory heats with different combinations of microalloying additions were produced in laboratory vacuum induction furnace with inert Ar atmosphere and deep vacuum. The main objective of the experimental heats was to determine the optimum microalloying additions for industrial trials on fixed alloying system. All three heats were Nb microalloyed, one was additionally alloyed with V and one with Ti. Logic behind the set-up composition was to examine if required mechanical properties could be achieved solely by Nb addition or if there is the need for V or Ti addition.

Steel was cast into 12 kg ingots and forget into 60 x 60 mm billets, which were then hot-rolled into 12 mm thick strips. Prior to rolling, billets were reheated to 1200 °C, which ensures full solubility of Nb(C,N) precipitates, to enable enhanced precipitation during the hot rolling. Precipitation phenomena was observed using calculated per pass mean-flow stresses, as it is described in paper [3]. Strips were air-cooled after hot rolling, followed by heat treatment consisting of water quenching and low-temperature tempering with air cooling.

Testing

Chemical composition was performed using optical emission spectroscopy (OES) ARL MA-310 for Si, Mn, Cr, Ni, Mo, Cu, Ti, Nb, V and B. Amount of C, S and N were determined with combustion method using LECO CS-600 for C and S and LECO TC-500 for N.

Specimens for evaluation of mechanical properties in quenched (Q) and QT condition were prepared, which included tensile test in accordance with EN ISO 6892-1 in longitudinal direction, and Charpy pendulum impact toughness (KV_2) at -40 °C in transverse direction in accordance with EN ISO 148-1.

Samples for metallographic examinations were prepared and Nital etched in order to compare prior austenite grain size (PAGS) and distribution between three experimental heats. Based on the results of mechanical testing and metallographic analysis, the most promising microalloying system was selected for further testing prior to industrial trials.

Samples for hot-compression testing on Gleeble 1500D were taken from plate and machined to cylindrical specimen with diameter and height of 10 and 15 mm, respectively. Plain hot compression and multi-stage tests were performed in accordance with scheme in Figure 1a and Figure 1b, respectively. Plain hot compression tests were conducted to study the dynamic recrystallization kinetics and the effect of thermomechanical parameters on grain size and distribution, in order to determine optimal parameters for hot rolling on industrial scale. Five-stage compression testing was done to determine the non-recrystallization temperature (T_{nr}). First the average flow stress for each deformation stage was determined and plotted against inverse of the absolute temperature. Intersection between linear regression of two distinct linear regions represents T_{nr}.

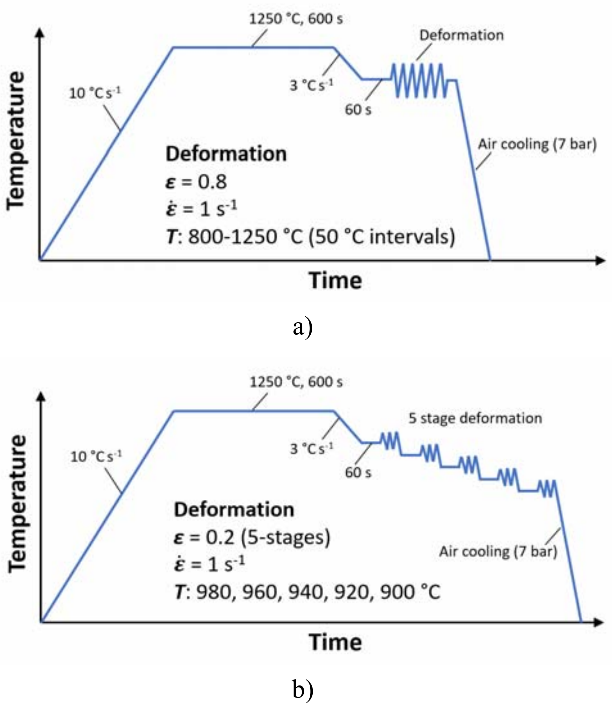


Figure 1. Schematic representation of a) plain hot compression test and b) multi-stage hot compression test.

Differential thermal analysis (DTA) was performed on cylindrical specimens with diameter and length of 4 and 3mm, respectively. Empty Al_2O_3 crucible cup was used as a reference. High-purity Ar 5.0 inert gas was used throughout the test to prevent oxidation. Three cycles of heating to 1550 °C and cooling to 1000 °C were performed, using heating/cooling rates of 20, 10 and 5 °C/min. Liquidus (T_{liq}) and solids temperature (T_{sol}) were determined on heating curves at 5 °C/min, using tangential method.

Dilatometric tests were done to study phase transitions in order to determine A_1, A_3 and M_s temperatures, as also the necessary cooling rate to achieve fully martensitic microstructure after water quenching. Cylindrical specimens of diameter and length of 3 and 10 mm were used, respectively. Samples were heated with heating rate 1 °C/s to austenitizing temperature 900 °C and held for 15 minutes for homogenisation, followed by cooling using varying cooling rates from 0,1 to 100 °C/s.

2.2 Industrial scale

Production process

Based on the results of laboratory testing, slightly modified chemical composition of Nb-Ti steel was selected for industrial trials. Steel scrap with limited copper content was charged and melted in electric arc furnace (EAF). Special care was taken for optimal dephosphorisation by strict temperature control, slag basicity and active oxygen content. Steel melt was tapped without carryover EAF slag to prevent P_2O_5 reduction.

Steel melt was fully killed and high basicity slag was formed with lime addition to ensure optimal cleanliness and low sulphur content. Prior to vacuum treatment, all alloying additions were added, slag composition and viscosity were adjusted and melt was reheated to correct temperature and homogenised using bottom Ar stirring. Vacuum degassing was conducted to reduce the hydrogen and nitrogen content. Due to intensive Ar stirring and high basicity slag, optimal conditions for desulphurisation were achieved, resulting in less than 10 ppm S in the final heat analysis. After degassing wires were injected to the melt, including CaSi cored wire for modification of non-metallic inclusions to ensure high cleanliness and prevent nozzle clogging due to clusters of Al_2O_3 inclusions.

Steel was cast into slabs on continuous caster, where special care was taken to prevent possible reoxidation. Based on T_{liq} determined on DTA, optimal casting temperature was chosen. Slabs were stack cooled prior to charging into reheating furnace and hot rolled into plates with thicknesses of 8, 10, and 15 mm. Rolling was divided into three phases, roughing, holding, and finishing. During roughing, the aim was to achieve grain refinement with static recrystallization between the passes. Holding was conducted so that finishing can be done in the temperature range where intense precipitation of Nb(C,N) occurs, resulting in refined PAGS. Plates were air cooled.

Last stage of the production process was the heat treatment consisting of water quenching after holding on austenitization temperature of 920 °C, which was selected based on results of dilatometric tests. Finally, low-temperature tempering at 180 °C was conducted.

Testing

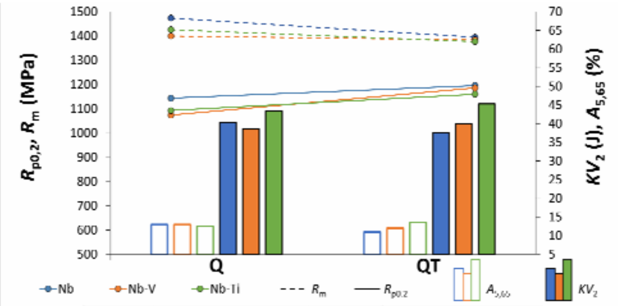
Heat analysis was determined using OES and LECO and mechanical properties were evaluated in QT in Q condition. Additionally, effect of holding on elevated temperatures on mechanical properties was studied.

3. RESULTS AND DISCUSSION

3.1 Laboratory scale

Chemical composition of laboratory heats is presented in Table 1. The amount of alloying elements is fixed, so that the effect of microalloying addition can be studied. Relatively small amounts of microalloying addition were added to prevent possible segregation, which can result in centreline formation of the Nb(C,N) eutectic [4]. Generally, alloying system C-Mn is set for sufficient strength, with additions of Cr, Mo, Ni and B for hardenability, Ni is also added for low-temperature toughness, as it improves dislocation mobility within the iron lattice, shifting ductile-to-brittle temperature to lower temperatures. Amount of Cu is increased to simulate the industrial condition where steel scrap is used, as Cu content is higher compared to iron ore-based steelmaking.

Mechanical properties in Q and QT condition in relation to microalloying elements used is presented in Figure 2. Increase in $R_{p0,2}$ and decrease in tensile strength (R_m) is observed after low-temperature tempering. All three microalloying systems achieve the minimum required $R_{p0,2}$ of 1100 MPa, as also KV_2 of 27 J at -40 °C in transverse direction. When it comes to elongation ($A_{5,65}$) and R_m, most of the producers guarantee $A_{5,65}$ of at least 10 %, which is the same as lower strength grade S960QL, whereas R_m is often in the range 1200-1550 MPa. In our case, $A_{5,65}$ of at least 11 % and R_m in the range between 1350 and 1400 MPa were achieved.



Steel	Condition	$R_{p0,2}$, MPa	R_m, MPa	$A_{5,65}$, %	KV_2, J
Nb	Q	1144	1474	13	40
	QT	1196	1395	11	39
Nb-V	Q	1074	1400	13	39
	QT	1186	1384	12	40
Nb-Ti	Q	1093	1426	13	43
	QT	1160	1377	14	45

Figure 2. Mechanical properties of laboratory S1100QL heats in Q and QT condition.

PAGS distribution, expressed as equivalent circle diameter (ECD) calculated from individual grain area, is presented in Figure 3. Results are plotted against measured area and displayed in 15 area classes, which yields optimal results regarding possible bimodality, according to [5]. It is evident that the finest PAGS and narrowest distribution were achieved by Nb-Ti microalloyed steel, which explains highest $A_{5,65}$ and KV_2 values. Finer PAGS is the consequence of Ti addition, which forms small and stable TiN that pin austenite grains during reheating prior to hot deformation. Due to the finest PAGS, Nb-Ti microalloyed steel was chosen for industrial testing.

Table 1. Chemical composition of the experimental heats in mass. %.

Heat	C	Si	Mn	Cr	Cu	Ni	Mo	Ti	Nb	V	B	N
Nb	0,19	0,24	0,85	0,52	0,38	1,3	0,42	-	0,013	-	0,0011	0,0029
Nb-V	0,17	0,23	0,88	0,51	0,37	1,3	0,40	-	0,013	0,02	0,0011	0,0046
Nb-Ti	0,18	0,23	0,87	0,52	0,35	1,3	0,41	0,013	0,014	-	0,0011	0,0031

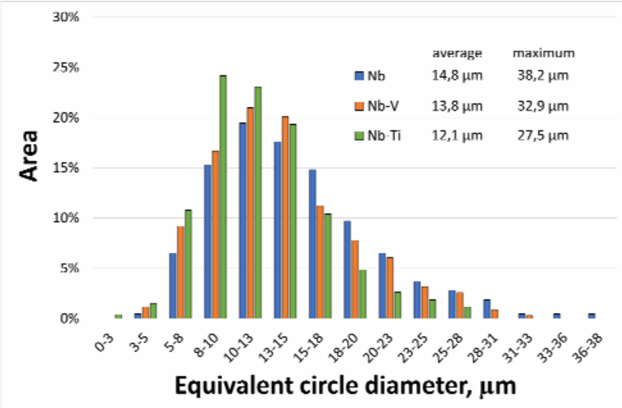


Figure 3. PAGS distribution in relation to microalloying system used.

For successful industrial implementation several parameters are critical, such as superheat before casting, thus T_{liq} of 1505 °C was determined from DTA heating curve. Optimal superheat was calculated to be 1530 °C. T_{sol} was determined to be 1473 °C.

Another critical process is hot rolling, where T_{nr} is crucial for the rolling schedule. It was determined to be 932 °C and practically matched the empirically determined T_{nr}, using widely accepted Boratto equation [6], calculated at 929 °C for a given chemical composition.

Results of the plain one-stage compressions tests, in terms of achieved microstructural evolution, are presented in Figure 4. PAGS increase with deformation temperature, due to enhanced mobility at elevated temperatures. First elongated grains are visible at 1000 °C, at 950 °C a combination of elongated and equiaxed grains is observed, while at 900 °C just a few equiaxed grains remain. Results are in accordance with calculated and experimentally determined T_{nr}.

For heat treatment, A_{c3}, A_{c1} and M_s temperatures were determined to be 800, 700 and 386 °C, respectively. Upper critical cooling rate, where fully martensitic microstructure is achieved, was determined to be 10 °C/s, which means, that water-quenching should result in martensitic microstructure, especially as the thicknesses of S1100QL plates are typically rather low.

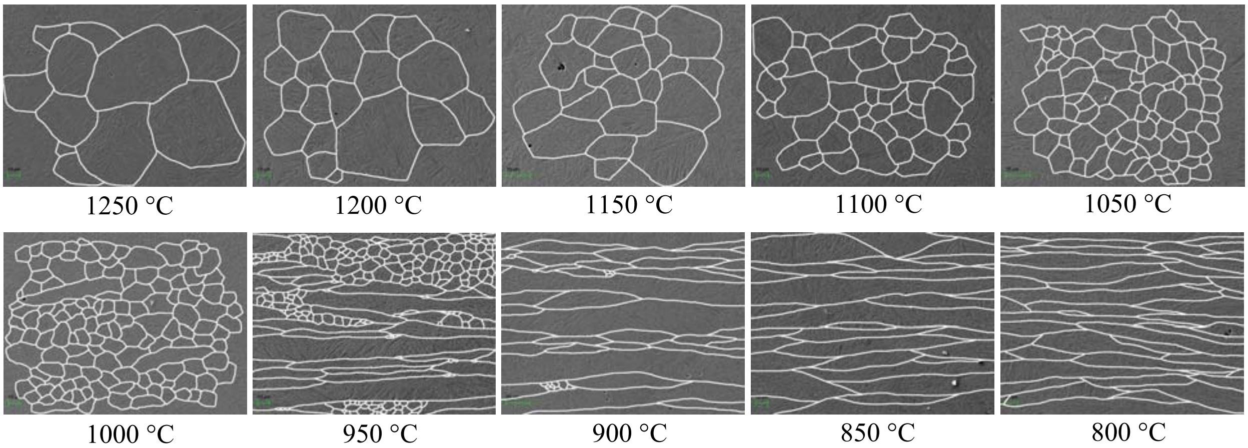


Figure 4. Evolution of microstructure in relation to deformation temperature of hot-compression tests.

3.2 Industrial scale

Heat analysis presented and compared to laboratory heat in Table 2. Some modifications were made, compared to laboratory heat, especially the increase of Mn and decrease of Ni content.

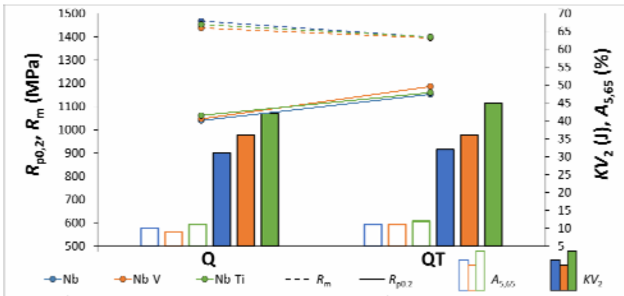
Quarto plates with thicknesses 8, 10 and 15 mm were produced as described in section 2.2. Microstructure in delivery QT condition is fully martensitic. Mechanical properties in Q and QT condition in relation to the plate thickness are presented in Figure 5. $R_{p0,2}$ increases with low-temperature tempering, providing a minimum of over 1100 MPa. All plates achieved over 10 % $A_{5,65}$ and KV_2 at least 27 J at -40 °C in transverse direction. KV_2 increases with plate thickness, however one needs to notice that subsized Charpy V-notch specimens 7,5x10x55 were used for 8 mm thick plate, compared to standard 10x10x55 specimens used for 10 and 15 mm thick plate. Overall, all mechanical properties meet the requirements for S1100QL grade. Low-temperature tempering had a major contribution, especially in $R_{p0,2}$ increase, which could be due to the first stage of tempering by fine transition carbides precipitation and stress relaxation [7], [8]. Due to tempering at low temperatures, S1100QL has enhanced abrasion resistance, compared to lower strength grades.

Table 2. Chemical composition of the industrial heat and comparison with laboratory heat in mass. %.

Heat	C	S	P	Si	Mn	Cr	Cu	Ni	Mo	Ti	Nb	B	N	CEV
Industrial	0,17	0,0005	0,006	0,23	1,21	0,53	0,17	1,0	0,47	0,025	0,026	0,0016	0,0045	0,65
Laboratory	0,18	0,0005	0,007	0,23	0,87	0,52	0,35	1,3	0,41	0,013	0,014	0,0011	0,0031	0,62

Carbon equivalent was calculated according to the equation (1) of International institute of welding IIW.

$$CEV = C + Mn/6 + (Cr+Mo+V)/5 + (Ni+Cu)/15 \quad (1)$$



Thickness, mm	Condition	$R_{p0,2}$, MPa	R_m, MPa	$A_{5,65}$, %	KV_2, J
8	Q	1040	1468	10	31
	QT	1153	1399	11	32
10	Q	1047	1438	9	36
	QT	1186	1395	11	40
15	Q	1006	1453	11	42
	QT	1160	1399	12	45

Figure 5. Mechanical properties of S1100QL plates in Q and QT condition.

S1100QL steel is often exposed to elevated temperatures, for example during preheating before welding. Figure 6 represents the effect of holding time at elevated temperatures on mechanical properties. KV_2 is stable up to 180 °C, which is the temperature used for low-temperature tempering. At 200 °C a drop occurs due to tempering embrittlement, typical for alloyed high-strength steels. Above 550 °C the KV_2 increases due to tempering of the martensite. $R_{p0,2}$ and R_m drop with increasing temperature, also the R_m to $R_{p0,2}$ ratio decreases. $A_{5,65}$ increases above 550 °C, similarly to KV_2. Overall, maximum temperature which should not be exceeded is 180 °C.

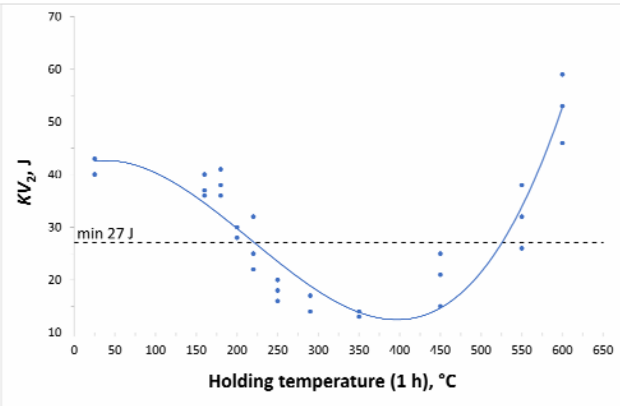


Figure 6. Effect of holding at elevated temperatures on impact toughness at -40 °C in transverse direction.

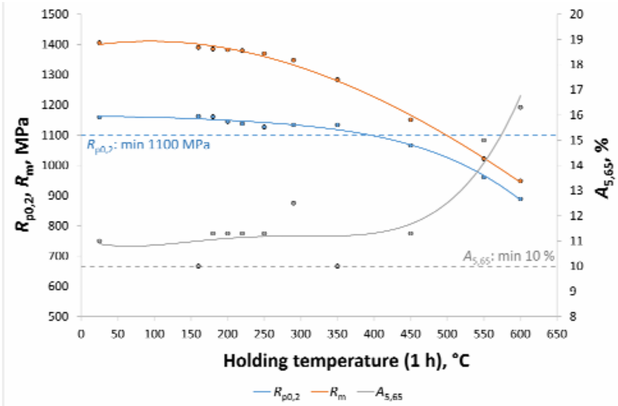


Figure 7. Effect of holding at elevated temperatures on tensile properties in transverse direction.

4. CONCLUSION

Laboratory test heats of ultra-high-strength S1100QL steel with different microalloying systems (Nb, Nb-V and Nb-Ti) were prepared and tested to determine optimal composition and processing parameters for industrial implementation. Based on the results, quarto plates were produced and tested to evaluate mechanical properties in final QT condition. The following results were obtained:

- Nb-Ti provided superior mechanical properties compared to Nb and Nb-V, especially in terms of KV_2 and $A_{5,65}$, due to pinning effect of small TiN precipitates, providing finer PAGS.
- Plates achieved the required mechanical properties in QT condition, where low-temperature tempering contributed to increase in $R_{p0,2}$, compared to Q condition.
- 180 °C was selected to be the maximum temperature which should not be exceeded as there is a drop of impact toughness, which occurs due to the tempering embrittlement.

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