

## NOVEL CONCEPT FOR DIRECT-QUENCHED ULTRAHIGH-STRENGTH STEEL

KAIJALAINEN Antti<sup>1</sup>, SAASTAMOINEN Ari<sup>1</sup>, HANNULA Jaakko<sup>1</sup>, HEIKKALA Jouko<sup>1</sup>, KESTI Vili<sup>2</sup>,  
SUIKKANEN Pasi<sup>2</sup>, PORTER David<sup>1</sup>, KÖMI Jukka<sup>1</sup>

<sup>1</sup>*Materials and Production Engineering - University of Oulu, Oulu, Finland, EU*

<sup>2</sup>*SSAB Europe Oy, Raahе, Finland, EU*

### Abstract

In the present study, direct-quenched steel with and without tempering at 600 °C has been studied in order to evaluate the possibilities of widening the range of strengths that can be produced from a single base composition. Microstructural studies of direct-quenched (DQ) and direct-quenched and tempered (DQT) steels showed that, in both cases, the microstructure consisted of mainly bainite formed from highly pancaked prior austenite grains, the main difference being a larger carbide size in the tempered condition. The results show that steels with minimum specified yield stresses of 900 and 960 MPa with good impact toughness and bendability properties can be produced from a single composition by employing respectively DQ and DQT processing. After tempering impact toughness and bendability was even better.

**Keywords:** Bainite, bendability, direct quenching, ductility, tempering

### 1. INTRODUCTION

Ultrahigh-strength strip steels, using low levels of carbon and other alloying elements, produced by thermomechanical controlled processing and direct quenching (TM-DQ) without tempering have become interesting materials for structural applications, since they cost effectively exhibit a good combination of mechanical properties, weldability [1, 2] and bendability [3]. However, tempering has been investigated to evaluate the possibilities of widening the range of strengths that can be produced from a single composition and tempering has been shown that this produces high strength combined with high toughness and good processing behavior [4].

In TMCP, austenite conditioning is an important processing stage affecting both strength and toughness [5]. Previous studies [6-9] have shown that an increase in total reduction ( $R_{tot}$ ) in the non-recrystallization regime of austenite in conjunction with a decrease in finish rolling temperature (FRT) produces a mixture of ferritic and granular bainitic microstructures near the strip surface together with a strong crystallographic texture. It is also well known that niobium and molybdenum increase the recrystallization temperature thereby enhancing the pancaking of austenite obtained at low FRTs.

Hannula et al. [10] have reported that additions of molybdenum and niobium increase the strength values significantly during tempering due to precipitation hardening. Molybdenum reduces the activity of carbon and nitrogen leading to less precipitation in austenite and a greater amount of solute niobium and molybdenum to form precipitates in ferrite during tempering. Similarly, vanadium has been shown to retard softening during tempering [11] and the yield strength of a V-alloyed steel increased during tempering due to significant precipitate strengthening [12].

The aim of the present paper is to report the effects of direct quenching with and without subsequent tempering on the mechanical, Charpy V and bendability properties of a single composition ultrahigh-strength steel when the austenite prior to quenching was highly deformed, i.e. had a high value of  $R_{tot}$ .

## 2. EXPERIMENTAL

### 2.1. Investigated materials

The experimental steel with the composition given in **Table 1** was thermomechanically rolled to an 8 mm strip thickness (*t*) with a finish rolling temperature (FRT) of *nd* thermomechanically rolled to *xxeruremnet* ~850 °C and direct quenched. To evaluate the possibilities of widening the range of strengths that can be produced from a single base composition, tempering experiments were performed by placing steel strips into a furnace previously heated to 600 °C. After reaching the peak temperature in 75 minutes, a soaking time of 30 minutes was applied to complete the heat treatment after which samples were cooled freely in air.

**Table 1** Chemical composition of experimental steel (in wt.%)

C	Mn	Si	Cr	Mo	Nb	V	B	Ti	Al
0.1	1.2	0.2	1.0	0.14	0.06	0.012	0.0012	0.023	0.04

### 2.2. Microstructural characterization

The prior austenite grain structure was studied using a laser scanning confocal microscope (LSCM) (VK-X200, Keyence Ltd) after picric acid etching [13]. Eq. (1) was used to determine the total rolling reduction below the recrystallization temperature ( $R_{tot}$ ) [14].

$$R_{tot} = \left( 1 - \sqrt{\frac{\bar{L}_{ND}}{\bar{L}_{RD}}} \right) \times 100\% \quad (1)$$

$\bar{L}_{RD}$  and  $\bar{L}_{ND}$  are grain boundary mean linear intercept lengths based on intercepts along the rolling direction and the strip normal direction. General characterization of the transformation microstructures was performed on Nital-etched specimens with a field emission scanning electron microscope (FESEM) (Ultra plus, Zeiss). Electron backscatter diffraction (EBSD) measurements (ODF texture) and analysis were performed using Oxford-HKL acquisition and analysis software. For the EBSD measurements the FESEM was operated at 15 kV and the step size was 0.2  $\mu\text{m}$ .

### 2.3. Mechanical testing

Microhardness was measured using a Micro-Hardness Tester (CSM) under a 1 N (HV0.1) load with ten measurements at eight depths below the surfaces and on the centerline. Macrohardness was measured using a Duramin-A300 (Struers) under a 100 N load (HV10) with five measurements at various depths. Tensile tests were carried out at room temperature in accordance with the European standard EN 10002 using flat specimens (8 x 20 x 120 mm<sup>3</sup>), cut with their axes both at 0° and 90° to the rolling direction (RD). Charpy V impact testing was conducted at -60 °C, using sub-size specimens of 7.5 x 10 x 55 mm<sup>3</sup> taken in both the longitudinal and transverse directions relative to the RD in accordance with EN ISO 148-1.

Three-point bending tests were carried out in an Ursviken Optima 100 bending machine to a bending angle of 90 degrees. Plate specimens, 8 x 300 x 300 mm<sup>3</sup>, were bent with their axes parallel to both the transverse and rolling directions. The die opening width (*W*) employed was 100 mm and the punch radii (*r*) ranged from 8 mm (*r/t* 1.0) to 20 mm (*r/t* 2.5). After bending, the quality of the bent surface was examined visually, as described in Ref. [15]. In the evaluation, the formation of a slight nut-shape did not lead to rejection, but if any surface waviness or more severe surface defects appeared, the bend test was considered as failed. The minimum bending radius was that resulting in a defect-free bend.

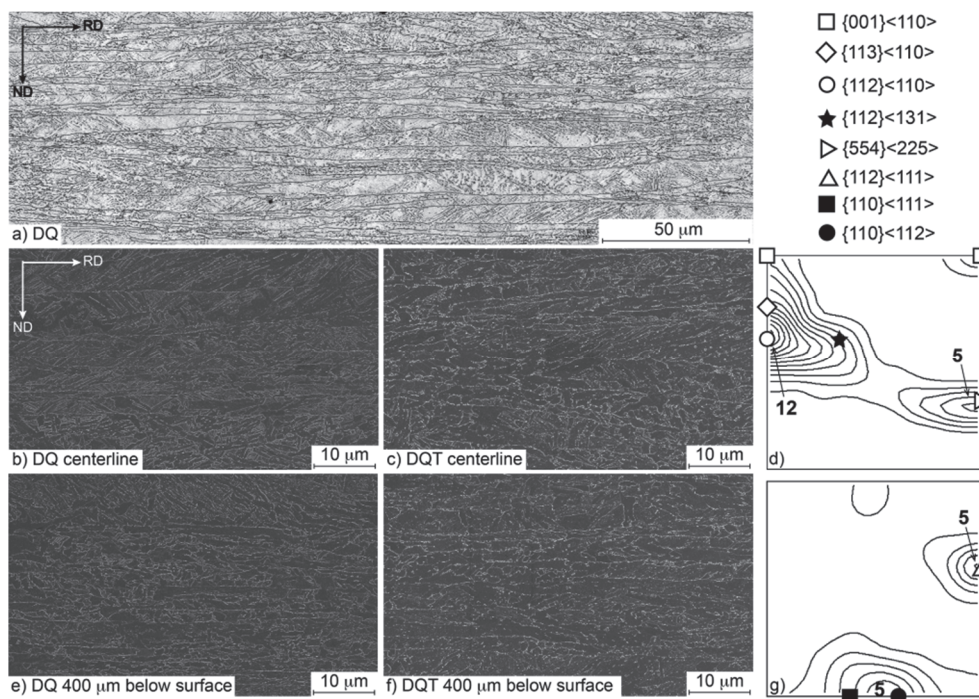
### 3. RESULTS AND DISCUSSION

#### 3.1. Microstructure

The influence of finish rolling below  $T_{NR}$  on the austenite morphology at the quarter-thickness in the DQ material is shown in **Figure 1a**.

The relatively low FRT gave a relatively large total reduction to the austenite below the recrystallization temperature ( $R_{tot}$ ), i.e. 69.3 % ( $\bar{L}_{RD}$  and  $\bar{L}_{ND}$  were  $3.0 (\pm 0.2) \mu\text{m}$  and  $32.3 (\pm 3.9) \mu\text{m}$ , respectively). Tempering did not affect these parameters.

The typical microstructures at the centerlines and the subsurface regions down to a depth of 500  $\mu\text{m}$  are given in **Figure 1**. The transformation microstructures at the centerline of the DQ specimen consisted of mixtures of upper and granular bainite and a small fraction of auto-tempered martensite (**Figure 1b**). The tempered specimen (**Figure 1c**) consisted of similar phases as the DQ state the carbide precipitates are coarser. Down to  $\sim 50 \mu\text{m}$  from the surface, the specimens consisted of polygonal ferrite interspersed among other carbon-rich microstructural components. This is also apparent in the microhardness profiles in **Figure 2b** where the hardness 50  $\mu\text{m}$  below the surface is as low as approx. 260HV. In the layer from 50  $\mu\text{m}$  to 400  $\mu\text{m}$ , specimens consist of granular and upper bainite (**Figure 1e**) however a small fraction of ferrite also occurs. The incidence of softer microstructures like ferrite and granular bainite formed due to the highly pancaked austenite as observed in previous studies [7].

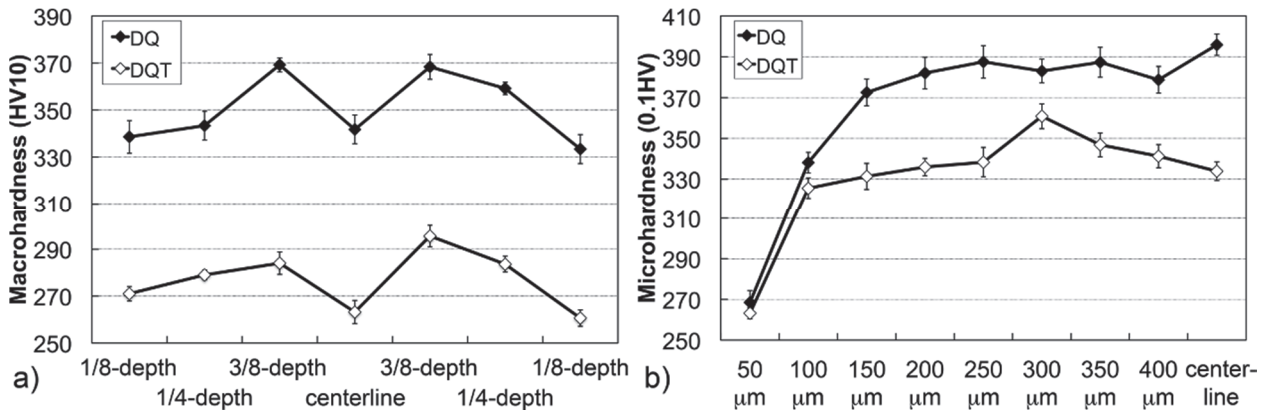


**Figure 1** (a) Prior austenite grain boundaries in DQ material at the quarter-thickness. Typical microstructures at the centerline of (b) DQ and (c) DQT and the surface of (e) DQ and (f) DQT after etching with 2 % Nital.

$\phi_2 = 45^\circ$  ODF sections illustrating the (d) centerline and (g) surface texture. (Levels: 1, 2, 3...)

The crystallographic texture of the DQ material showed strong  $\sim\{112\}<110 - 131>$  and  $\sim\{554\}<225>$  texture components at the centerline (**Figure 1d**) and minor  $\sim\{112\}<111>$  and  $\{110\}<111 - 112>$  components near the surface (**Figure 1g**). Lath and effective grain sizes on the centerline of the DQ material were  $1.28 (\pm 0.02) \mu\text{m}$  and  $1.91 (\pm 0.07) \mu\text{m}$  respectively. Lath sizes are equivalent circle diameters (ECD) for regions bound by low-angle boundaries, in the range  $2.5 - 15^\circ$ , while effective grain sizes are ECD values for regions bound by high-angle boundaries,  $>15^\circ$ . These lath and grain sizes were coarser than found previously (lath  $0.8-1.0 \mu\text{m}$  and

effective grain size 1.1-1.3  $\mu\text{m}$ ) [6], but this is because the present microstructures contain more granular bainite with correspondingly lower hardness at the centerline (**Figure 2**). Tempering had neither effect on crystallographic texture nor effective grain size.



**Figure 2** Mean values of (a) a macrohardness and (b) microhardness at various depth below the surface

### 3.2. Mechanical properties

The mechanical and bendability properties of the materials are presented in **Table 2** and a few examples of bends are shown in **Figure 3**. The bendability results are grouped such that data for the bend axis parallel to transverse direction are located in the columns showing longitudinal tensile properties (and vice versa) since both cases lead to principal strains in the same direction. It can be seen that for the longitudinal test direction the investigated ultrahigh-strength steels have yield strengths ( $R_{p0.2}$ ) of 1084 MPa and 890 MPa (DQ and DQT, respectively), and tensile strengths ( $R_m$ ) of 1110 MPa and 920 MPa, respectively. The total elongation to fracture ( $A_5$ ) is slightly higher for the longitudinal specimens in the DQT state, i.e. 14.2 %, as compared to 11.1 % in the DQ state. For comparison with the hardnesses, **Table 3** also includes the mean macrohardness of the materials as measured on the RD-ND cross-sections taken from **Figure 2a**.

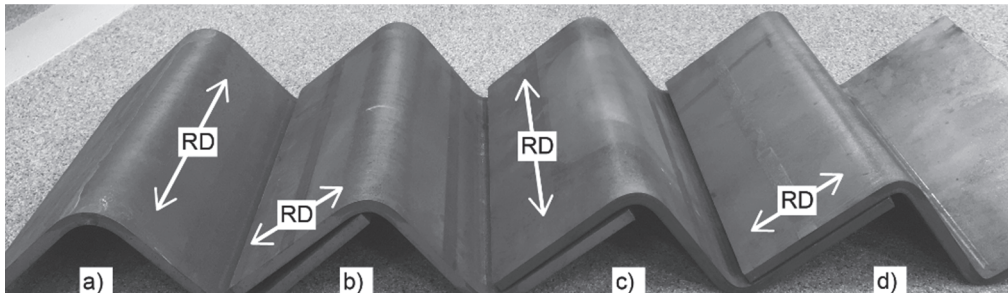
**Table 2** Mechanical and bendability properties of investigated materials

	Longitudinal		Transverse	
	DQ	DQT	DQ	DQ-T
Yield strength ( $R_{p0.2}$ , MPa)	1084	890	1032	932
Tensile strength ( $R_m$ , MPa)	1110	920	1101	956
Total elongation ( $A_5$ , %)	11.1	14.2	9.3	12.9
Uniform elongation ( $A_g$ , %)	1.5	4.9	1.5	4.6
Through-thickness mean HV10	351	277	351	277
Impact toughness at -60 °C ( $\text{J}/\text{cm}^2$ )	64.2	105.0	74.6	78.3
Minimum bending radius [mm, ( $r/t$ )]	15 (1.9)	8 (1.0)	20 (2.5)	12 (1.5)
	bend axis perpendicular to RD <sup>1</sup>		bend axis parallel to RD <sup>1</sup>	

<sup>1</sup> Bending with the bend axis perpendicular to the RD induces principle strains in the longitudinal direction and vice versa.

Impact toughness at -60 °C was 64  $\text{J}/\text{cm}^2$  for longitudinal specimens and 74  $\text{J}/\text{cm}^2$  for transverse specimens in the DQ state, and after tempering impact toughness was slightly better. The minimum bending radius in DQ material was 2.5 times the sheet thickness (2.5t) in with the bend axis in the longitudinal direction (**Figure 3a**) and 1.9t transverse to the rolling direction (**Figure 3b**). In the DQT state, bendability was even better, and

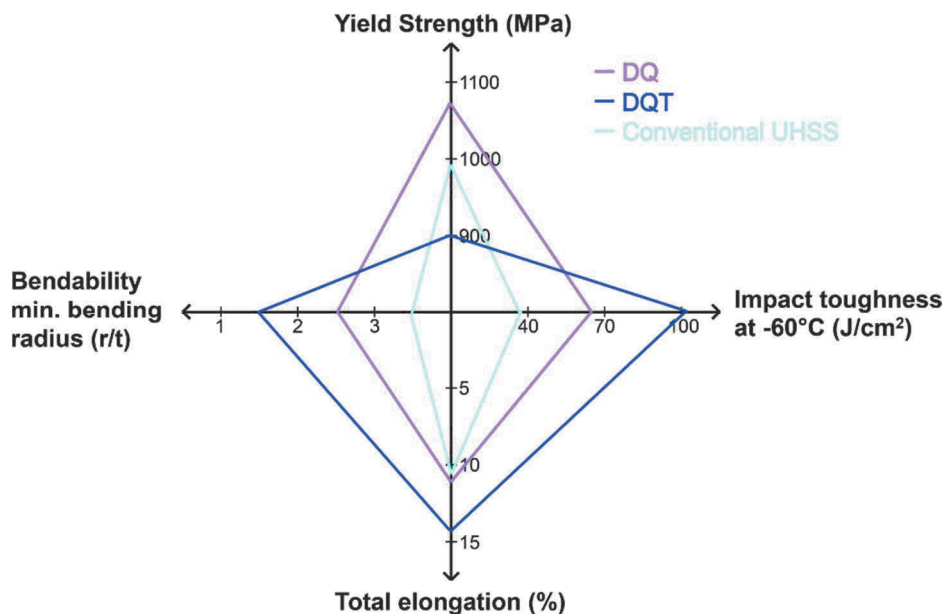
corresponding minimum bending radii were 1.5t (**Figure 3c**) and 1.0t (**Figure 3d**). For comparison, at the 900 MPa yield strength level is concerned, the bendability can be considered as being very good when the minimum usable bending radius is less than 3.0t.



**Figure 3** Examples of the minimum bending radius of the bent samples at a bending angle of 90 deg: (a) r20 (2.5t) bend axis along the rolling direction of DQ, (b) r15 (1.9t) bend axis transverse to the RD of DQ, (c) r12 (1.5t) bend line along the RD of DQT, and (d) r8 (1t) bend axis transverse to the RD of DQT

### 3.3. Relationship of microstructure and tempering on the mechanical properties

There are several ways of controlling the strength - toughness - formability balance in these direct-quenched UHSS. Additions of microalloying (Nb, Mo and V) enhanced desirable strength and improved the toughness properties by promoting the formation of pancaked austenite. Similarly, exceptionally good bendability is achieved with a soft subsurface microstructure (i.e. granular bainite) and the absence of an intense  $\sim\{112\}<111>$  texture component at the surface. The good toughness properties are the result of extensive pancaking, which produced intense  $\sim\{112\}<110 - 131>$  and  $\sim\{554\}<225>$  components at the expense of the  $\{100\}$  texture that facilitates cleavage cracking [16]. In the DQT state, Nb, Mo and V microalloying additions help retain a fine carbide structure and the dislocation structure achieved by direct quenching, which lead to excellent combinations of strength and toughness.



**Figure 4** Balance of strength, toughness, elongation to fracture and minimum bending radius of DQ, DQT and conventional UHSS

As can be seen from **Figure 4**, DQ and DQT materials provide excellent overall properties. The toughness and bendability properties of DQT material can be considered to be abnormally good at the 900 MPa yield

strength level concerned. Typically, the bendability of steel with a similar strength level is over 3 times the material thickness, whereas in the case of the DQT material in our studies it is 1.5 x t.

#### 4. CONCLUSION

In this work, it has been shown that highly pancaked prior austenite enhanced the formation of granular bainite in the strip subsurface layers even though the inner part of the strip was bainitic, providing an ultrahigh yield strength in excess of 960 MPa with excellent impact toughness and bendability in DQ state. Furthermore, tempering at 600 °C improved the toughness and bendability, while the yield strength decreased slightly to 900 MPa. Further research into the effects of chemical composition and tempering parameters will help to optimize the production parameters that best allow a single composition to be used for both DQ and DQT products.

#### ACKNOWLEDGEMENTS

***This work was made as a part of the Breakthrough Steels and Applications program of DIMECC Oy (the Digital, Internet, Materials & Engineering Co-Creation). The financial support of The Finnish Funding Agency for Technology and Innovation (Tekes) is gratefully acknowledged.***

#### REFERENCES

- [1] ASAHI, H., TSURU, E., HARA, T., SUGIYAMA, M., TERADA, Y., SHINADA, H., et al. Pipe production technology and properties of X120 linepipe. *Int. J. Offshore Polar Eng.* 2004, vol. 14, pp. 36-41.
- [2] HEMMILÄ, M., LAITINEN, R., LIIMATAINEN, T., PORTER D.A. Mechanical and technological properties of ultra high strength Optim steels. In: *Proc. 1st Int. Conf. "Super-High Strength Steels"*. Rome, 2005.
- [3] KESTI, V., KAIJALAINEN, A., VÄISÄNEN, A., JÄRVENPÄÄ, A., MÄÄTTÄ, A., AROLA, A.M., et al. Bendability and microstructure of direct quenched Optim® 960QC. *Mater. Sci. Forum.* 2014 vol. 783-786, pp. 818-824.
- [4] MOHRBACHER, H. Mo and Nb Alloying in Plate Steels for High Performance Applications. In: *Int. Symp. Recent Dev. Plate Steels*, Colorado, 2011, pp. 169-181.
- [5] TAMURA, I., OUCHI, C., TANAKA, T., SEKINE, H. *Thermomechanical processing of high strength low alloy steels*. Cornwall: Butterworth & Co. Ltd., 1988.
- [6] KAIJALAINEN, A.J., SUIKKANEN, P.P., LIMNELL, T.J., KARJALAINEN, L.P., KÖMI, J.I., PORTER, D.A. Effect of austenite grain structure on the strength and toughness of direct-quenched martensite. *J. Alloys Compd.* 2013, vol. 577, pp. S642-S648.
- [7] KAIJALAINEN, A.J., LIIMATAINEN, M., KESTI, V., HEIKKALA, J., LIIMATAINEN, T., PORTER, D.A. Influence of composition and hot rolling on the subsurface microstructure and bendability of ultrahigh-strength strip. *Metall. Mater. Trans. A.* 2016, vol. 47, pp. 4175-4188.
- [8] KAIJALAINEN, A.J., SUIKKANEN, P.P., KARJALAINEN, L.P., PORTER, D.A. Influence of subsurface microstructure on the bendability of ultrahigh-strength strip steel. *Mater. Sci. Eng. A.* 2016, vol. 654, pp. 151-160.
- [9] KAIJALAINEN, A.J., SUIKKANEN, P., KARJALAINEN, L.P., JONAS, J.J. Effect of austenite pancaking on the microstructure, texture, and bendability of an ultrahigh-strength strip steel. *Metall. Mater. Trans. A.* 2014, vol. 45, pp. 1273-1283.
- [10] HANNULA, J., PORTER, D.A., SOMANI, M.C., MEHTONEN, S. Effect of molybdenum and niobium on the microstructures and mechanical properties of direct-quenched high-strength steel. In: *5th Int. Conf. Thermomechanical Process.* Milan, 2016, pp. 5045.
- [11] SAASTAMOINEN, A., PORTER, D., SUIKKANEN, P. The microstructure and properties of direct quenched martensite subjected to both slow furnace tempering and rapid induction tempering cycles. In: *9th Int. Conf. Roll.* Venice, 2013.
- [12] BHADESHIA, H.K.D.H., HONEYCOMBE, S.R. *Steels. 3rd ed.* 2006. pp. 183-208.

- [13] BROWNRIGG, A., CURCIO, P., BOELEN, R. Etching of prior austenite grain boundaries in martensite. *Metallography*, 1975, vol. 8, pp. 529-533.
- [14] HIGGINSON, R.L., SELLARS, C.M. *Worked Examples in Quantitative Metallography*. London: Maney, 2003.
- [15] HEIKKALA, J., VÄISÄNEN, A. Usability testing of ultra high-strength steels. In: *Proc. 11th Bienn. Conf. Eng. Syst. Des. Anal.*, 2012, pp. 1-13.
- [16] YAN, P., GÜNGÖR, Ö.E., THIBAU P., LIEBEHERR, M., BHADESHIA, H.K.D.H. Tackling the toughness of steel pipes produced by high frequency induction welding and heat-treatment. *Mater. Sci. Eng. A*, 2011, vol. 528, pp. 8492-8499.