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OPTIMISATION OF THE Q-P PROCESS PARAMETERS FOR LOW ALLOYED STEELS WITH 0.2% C

OBRÓBKA Q-P DLA STALI O ZAWARTOŚCI 0.2% C

In steels which are treated by the quenching and partitioning (Q&P) process, carbon content is one of the crucial parameters because carbon contributes greatly to stabilization of retained austenite and strengthens the material. In the present study, the Q&P process was gradually optimised for two low-alloyed steels with 0.2% carbon content and with and without Cr addition. The results show that the cooling rate, as well as the austenitizing temperature, has a pronounced effect on microstructure evolution. The strength and elongation in the Mn, Si and Cr-alloyed steel was approx. 900 MPa and more than 30%, respectively.
Keywords: Q-P process, retained austenite (RA), AHSS, carbon content

W stalach poddanych obróbce hartowania i partycjonowania (*Quenching and Partitioning* – Q&P), zawartość węgla jest jednym z kluczowych parametrów, ponieważ węgiel znacząco wpływa na stabilizację austenitu szczątkowego i umacnia materiał. W niniejszych badaniach obróbka Q&P była stopniowo optymalizowana dla dwóch niskostopniowych stali o zawartości węgla 0,2% zawierających dodatek chromu oraz bez tego dodatku. Wyniki pokazują wyraźny wpływ szybkości chłodzenia oraz temperatury austenityzacji na ewolucję mikrostruktury. Wytrzymałość na rozciąganie oraz wydłużenie do zerwania w manganowo-krzemowo-chromowej stali wyniosły odpowiednio ok.900 MPa oraz ponad 30%.

1. Introduction

The carbon content in high-strength steels has a substantial influence on their mechanical properties, as it greatly affects the resulting type of microstructure. It is the governing aspect for selection of thermomechanical treatment parameters. The treatment of high-strength steels typically consists of unconventional procedures which deliver high ultimate strength combined with adequate ductility levels [1]. One of the available techniques for treating these advanced steels with low levels of alloying elements is the so-called Q&P process.

In the Q&P process, the carbon level effectively controls the formation of martensite and the stabilization of retained austenite. The resulting microstructure consists of martensite and foil-like retained austenite. The process was first described in 2003 [2].

Q&P process is a two-stage heat treating procedure which consists of full austenitization and rapid cooling down to a region between the M_s and M_f temperatures [3]. Its key parameters include the quenching temperature and the partitioning temperature, both being governed by the carbon content in steel [4] (Fig. 1). With increasing carbon content, the quenching temperature decreases, opening the possibility of stabilizing a greater portion of austenite. By contrast, with decreasing carbon level the temperature interval for stabilization expands considerably.

Another important aspect in obtaining the desired microstructure is the chemistry of the steel. It should suppress carbide formation and pearlitic and bainitic transformations, while promoting migration of carbon to austenite. By these processes, austenite becomes stable [5-6].

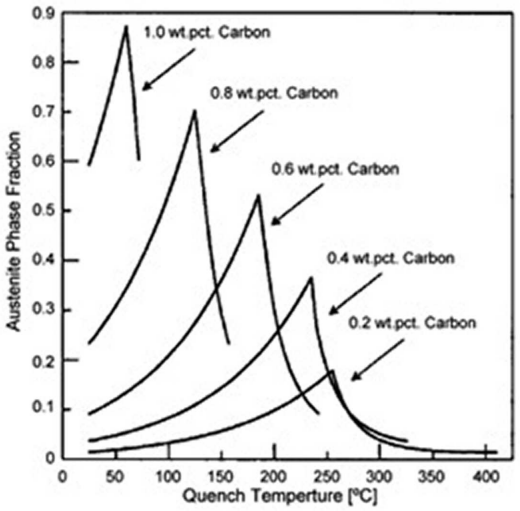

Fig. 1. Expected RA fractions at various carbon levels [4]

2. Experimental programme

In the present experimental programme, parameters of the Q&P process were optimized for two low-alloyed steels, CMn-Si and CCrMnSi, with the carbon content of approx. 0.2%. The purpose of the effort was to explore the potential for treating low-carbon steels of this kind using the Q&P process.

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TABLE 1

Chemical composition of experimental steels [wt. %]

Steel	C	Mn	Si	P	S	Cr	Ni	Cu	Al	Nb	Mo
CMnSi	0.21	1.449	1.797	0.008	0.005	0.008	0.072	0.058	0.006	0.059	0.02
CCrMnSi	0.19	0.59	1.92	0.009	0.004	0.79	0.03	0.02	0.001	0.04	0.03

For maximum accuracy of process parameters, the treatment was performed in a thermomechanical simulator. Thanks to its combined electrical resistance and induction heating principle, the device achieves heating rates up to 500°C/s. It also offers a wide range of cooling rates, reaching up to 250°C/s in steels.

Metallographic observation was performed using light (LM) and scanning electron microscopes (SEM). The amount of retained austenite was measured by means of XRD phase analysis in the automatic powder diffractometer AXS Bruker D8 Discover with a position-sensitive area HI-STAR detector and a cobalt X-ray source ($\lambda - K\alpha$= 0.1790307 nm). Mechanical properties were mapped through HV10 hardness measurement and by tension testing on miniature specimens with 2×1.2 mm cross-section and a gauge length of 5 mm.

The carbon content in both steels was around 0.2%, which is a very low level when compared with steels treated by Q&P process up to now. Steels which are normally treated using this method contain more than 0.4% carbon [7-9]. The CMnSi steel was alloyed with Mn and Si to promote stabilization of retained austenite, solid solution strengthening and to suppress formation of carbides and pearlite [10]. In an effort to increase strength, hardenability and to suppress bainitic transformation, the CCrMnSi steel also contained an addition of Cr (Table 1).

The initial structures of steels contained ferrite and pearlite with the hardness value of 193 HV10 in CMnSi and 191 HV10 in CCrMnSi steel. The tensile strength was equal to 623 MPa with the ductility of 42% in the steel CMnSi and 658 MPa with the ductility A_{5mm} of 45% in the CCrMnSi steel.

2.1. Determination of phase transformation temperatures

If the desired microstructure, i.e. martensite and foil-like retained austenite along boundaries of martensite needles, is to be obtained in steels treated by Q&P process, the correct chemistries as well as processing parameters have to be found. Crucial parameters include the quenching temperature and the partitioning temperature which should lie between the M_s and M_f. The M_s and M_f were therefore measured using different methods. CCT and TTT diagrams were constructed using data computed with JMatPro program and the Ms temperature was calculated via Andrews phenomenological model [11] (Table 2). The temperatures found this way were then validated by means of dilatometric measurement using the cooling rate of 20°C/s. Bähr dilatometer was used with specimens of 5 mm diameter and 10 mm length.

TABLE 2

Transformation temperatures determined by various methods

Steel	JMatPro			Dilatometer - 20°C/s	Andrews
	M_s [°C]	M_f [°C]	A_{r3} [°C]	M_s [°C]	M_s [°C]
CMnSi	370	257	864	390	387
CCrMnSi	395	283	909	376	407

In CMnSi steel, the M_s temperature was between 370 and 390°C, depending on the method used. The largest discrepancy was found between the JMatPro calculation and the dilatometric measurement. In the CCrMnSi steel with lower Mn content and a chromium addition, the M_s temperature was higher, in the 395-407°C interval (TABLE 2).

2.2. Design of Q&P Process (QP)

In the effort to optimize the Q&P process parameters, a total of six different schedules were trialled on both experimental steels (TABLE 3). The QP1 schedule comprised heating to and soaking at 950°C for 100 s. Incremental deformation with the strain φ=5 was applied between 950 and 720°C. The quenching temperature (QT) was set at 300°C s. The holding time at this temperature was 10 s. For both steel, the quenching temperature was set between the M_s and M_f. The holding was followed by heating to the partitioning temperature (PT) of 350°C and another hold for 600 s. In order to compare the effects of incremental deformation on mechanical properties, a schedule with no deformation was carried out as well (QP2).

In addition, the impact of the rate of cooling from the soaking temperature to the quenching temperature was studied. In the QP3 schedule, the heating rate was increased from 17°C/s to 30°C/s. In the QP4 schedule, the effects of the quenching temperature were explored: the quenching temperature was reduced from the initial 300°C to 250°C.

As the austenitizing temperature has a pronounced effect on the microstructure evolution as well, the QP5 schedule involved an increased austenitizing temperature (from 950°C to 1000°C) with identical holding time of 100 s. In the last schedule, the rate of cooling from the austenitization temperature to the quenching temperature was increased as well: from 30 to 50°C/s (QP6) (TABLE 3).

TABLE 3

Q&P process parameters for both experimental materials

		TA/tA [°C/s]	No. of def. steps	Deform. process. window [°C]	Cooling rate [°C/s]	QT/Qt [°C/s]	PT/Pt [°C/s]
CMnSi	QP1	950/100s	20	950-720	17	300/10	350/600
	QP2		–	–			
	QP3		20	950-720	30		
	QP4		20	950-720	30	250/10	300/600
	QP5	1000/100	20	950-720	30	250/10	300/600
	QP6		20	950-720	50	250/10	300/600
CCrMnSi	QP1	950/100s	20	950-720	17	300/10	350/600
	QP2		–	–			
	QP3		20	950-720	30		
	QP4		20	950-720	30	250/10	300/600
	QP5	1000/100	20	950-720	30	250/10	300/600
	QP6		20	950-720	50	250/10	300/600

3. Results and Discussion

3.1. CMnSi Steel

The QP1 schedule with the soaking temperature of 950°C, 20 deformation steps and a quenching temperature of 300°C was the initial reference schedule for the optimization of Q&P processing. The resulting microstructure consisted of ferrite, coarse martensite-bainite blocks and retained austenite in an amount of 9%. The resulting hardness was 256 HV10 (Fig. 2, TABLE 4). The ultimate strength reached 835 MPa and the elongation was A_{5mm} = 37%.

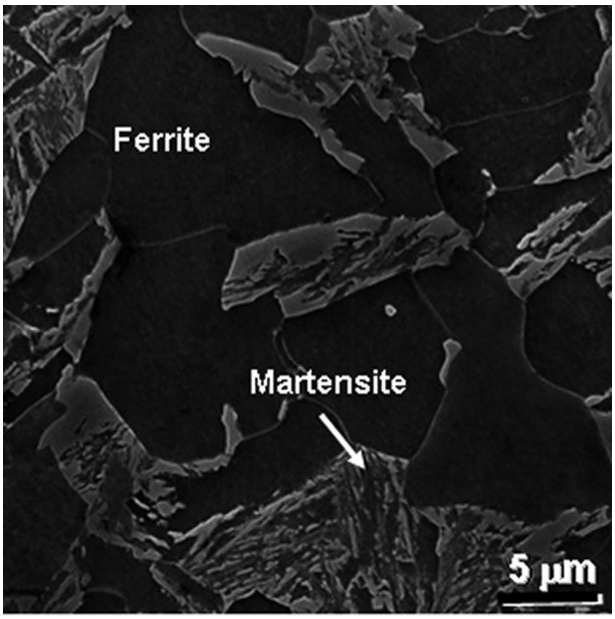

Fig. 2. CMnSi: 950°C/100 s – 20×def. – 350°C/600 s with cooling rate of 17°C/s (QP1) – Nital etch, SEM

Where incremental deformation was omitted, such as in the QP2 schedule, the resulting microstructure contained even coarser martensite-bainite islands, which did not have any effect on mechanical properties. As the desired microstructure with prevailing martensite was not achieved, a schedule with an increased cooling rate of 30°C/s was trialled (QP3). The result was a mixture of bainite, martensite, ferrite and retained austenite.

To promote martensite formation, the quenching temperature was reduced from 300°C to 250°C, and thus the partitioning temperature was reduced from 350°C to 300°C (QP4). The resulting microstructure was a mixture of ferrite, martensite, small fraction of bainite and 10% of retained austenite (Fig. 3). Quenching at a lower temperature did not lead to higher strength. The ultimate strength was 829 MPa and elongation reached 34% (TABLE 4).

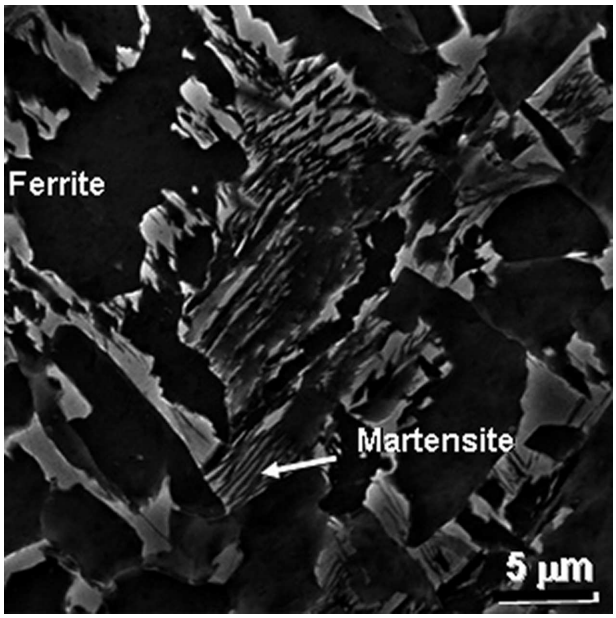

Fig. 3. CMnSi: 950°C/100 s – 20× def. – 300°C/600 s with cooling rate of 30°C/s (QP4) – Nital etch, SEM

Two-stage etching procedure was employed to find the distribution of retained austenite in the microstructure (step 1:

Nital, step 2: 10% water solution of $Na_2S_2O_5$). Retained austenite was found to exist in both granular form between ferrite grains and as foils within martensitic-bainitic islands (Fig. 4). Those were M-A constituent-type areas, which means they consisted of martensite and austenite.

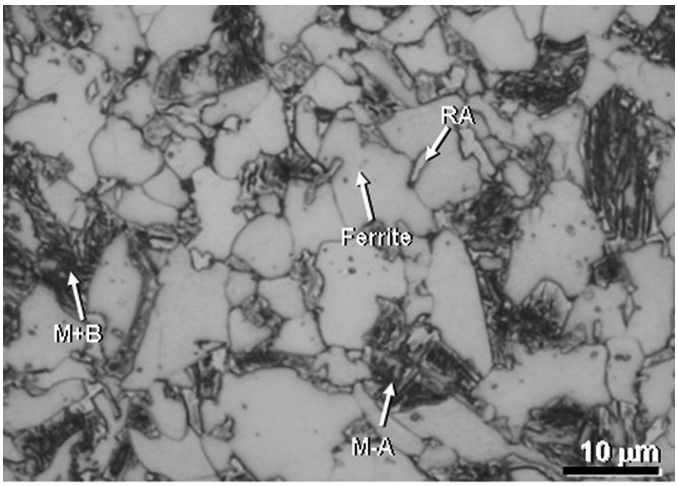

Fig. 4. CMnSi: 950°C/100 s – 20× def. – 300°C/600 s with the cooling rate of 30°C/s (QP4), two-stage etching, light micrograph

Raising the austenitizing temperature to 1000°C led to a higher martensite fraction and to an increase in hardness from the previous 244 HV10 to 283 HV10 (TABLE 4). This was reflected in both the increase in the retained austenite volume fraction to 15%, and the rise in strength to 889 MPa. The decline in the amount of ductile ferrite caused elongation to decrease to 25%.

TABLE 4
Results of mechanical tests for both experimental materials

		R_m [MPa]	A_{5mm} [%]	HV10 [-]	RA [%]
CMnSi	QP1	835	37	256	9
	QP2	834	36	259	7
	QP3	770	33	262	12
	QP4	829	34	244	10
	QP5	889	25	283	15
	QP6	1012	19	344	12
CCrMnSi	QP1	833	41	263	10
	QP2	873	34	271	8
	QP3	871	31	285	7
	QP4	891	32	276	14
	QP5	982	13	334	9
	QP6	1263	10	396	14

A further increase in the cooling rate to 50°C/s (applied in the QP6 schedule) promoted martensite formation and led to an even higher hardness of 344 HV10 (Fig. 5). Using this schedule, the highest strength in this steel was achieved: 1012 MPa at the elongation level of 19% (TABLE 4).

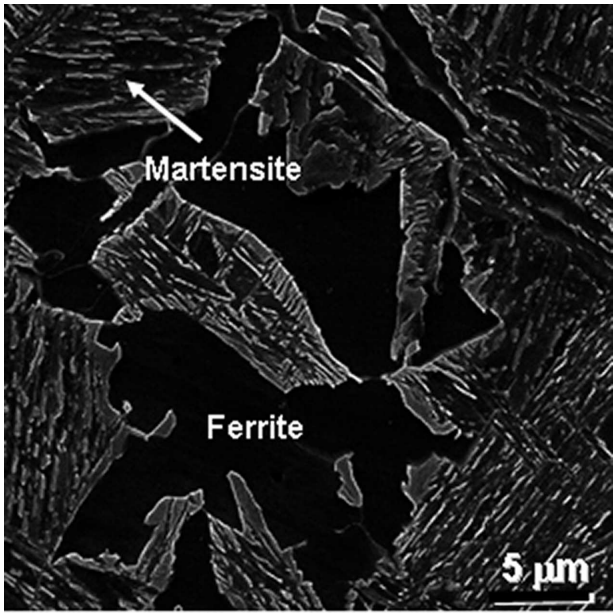

Fig. 5. CMnSi: 1000°C/100 s – 20× def. – 300°C/600 s with cooling rate of 50°C/s (QP6) – Nital etch, SEM

3.2. CCrMnSi Steel

The CCrMnSi steel with a higher chromium level was processed using the same Q&P schedules as those used for CMnSi steel (TABLE 3). The initial QP1 schedule led to a mixed microstructure of ferrite and martensite islands with a small fraction of bainite. 10% retained austenite was found in the microstructure (Fig. 6). The strength level of 833 MPa at an elongation of 41% is very close to the values of the CMnSi steel (TABLE 3).

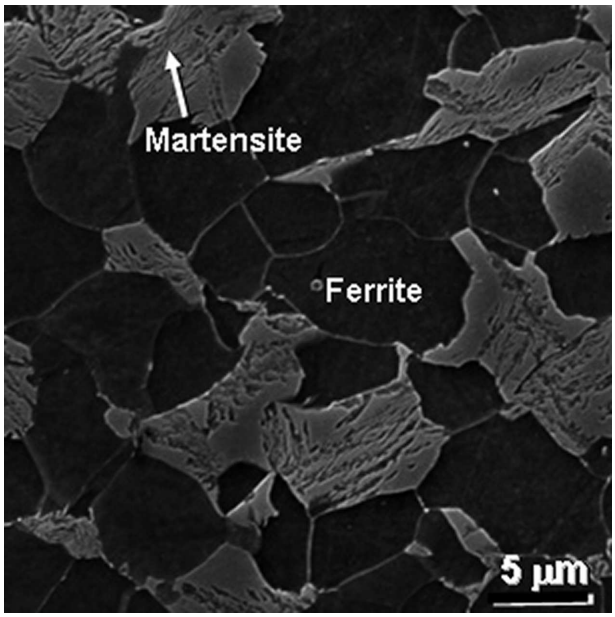

Fig. 6. CCrMnSi: 950°C/100 s – 20× def. – 350°C/600 s with cooling rate of 14°C/s (QP1) – Nital etch, SEM

The increase in strength was achieved by the accelerated cooling: from the initial 17°C/s to 30°C/s (QP3). The resulting microstructure showed no substantial differences in terms of

the distribution and morphology of microstructure constituents (Fig. 7).

However, the strength level was 871 MPa and the elongation was 34%. The addition of chromium did contribute to the increase in strength by 100 MPa over that of the CMnSi but it failed to produce predominantly martensitic structure without bainite and ferrite.

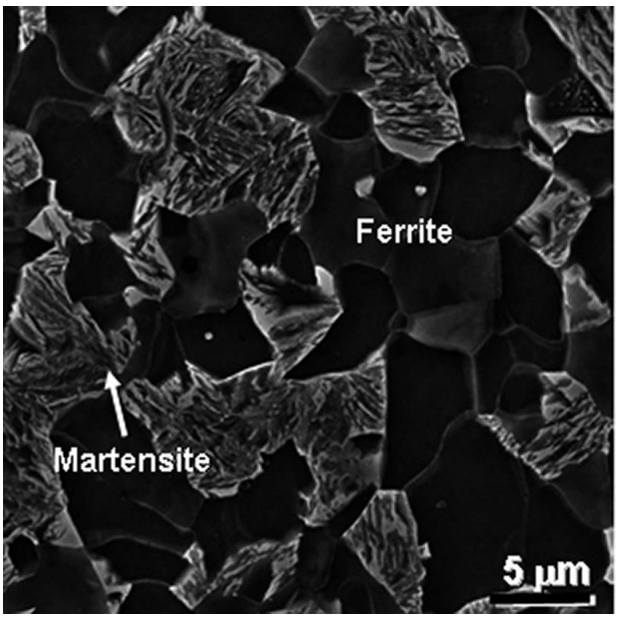

Fig. 7. CCrMnSi: 950°C/100 s – 20× def. – 350°C/600 s with cooling rate of 30°C/s (QP3) – Nital etch, SEM

A further reduction in the quenching temperature from the initial 300°C to 250°C caused the resulting strength to increase (QP4). The ultimate strength was 891 MPa and the elongation reached 32%. The retained austenite volume fraction was 14% (TABLE 4). Its distribution was found by two-stage etching. Retained austenite was found to be present in both globular form and in martensite-bainite islands (Fig. 8).

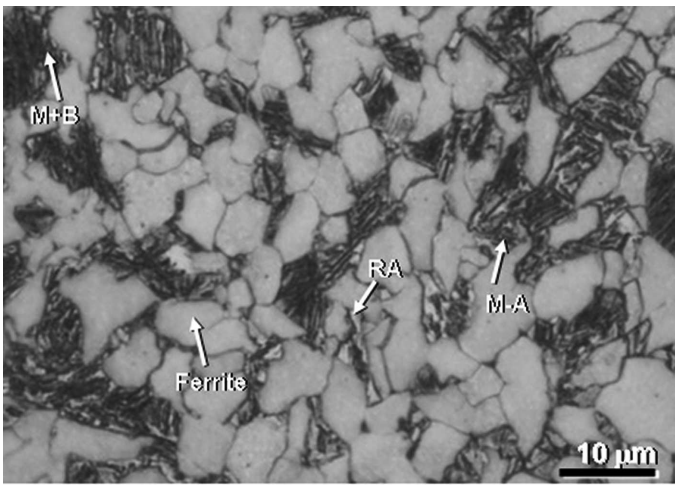

Fig. 8. CCrMnSi: 950°C/100 s – 20× def. – 300°C/600 s with the cooling rate of 30°C/s (QP4), two-stage etching, light micrograph

QP5 and QP6 schedules, in which the austenitizing temperature was raised to 1000°C, produced notably higher fractions of martensite (Fig. 9). When the cooling rate was concurrently decreased from 30°C/s to 50°C/s (QP6), the resulting material contained a large amount of martensite and 14% retained austenite (TABLE 4). Hardness reached almost 400 HV10. The change in the microstructure composition was reflected in the considerable increase in strength to 1263 MPa at an elongation of 10%.

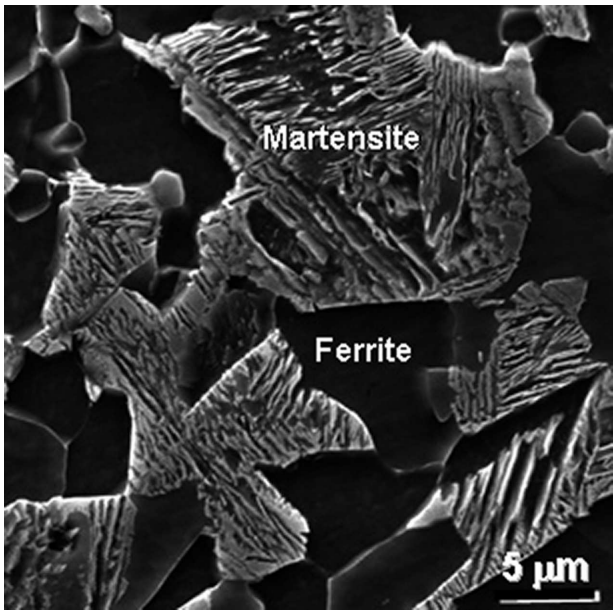

Fig. 9. CCrMnSi: 1000°C/100 s – 20× def. – 300°C/600 s with the cooling rate of 50°C/s (QP6) – Nital, SEM

In this schedule, the chromium addition had a strong effect. Unlike the CMnSi steel which lacked Cr, this material showed an increase in strength by 251 MPa accompanied by a decline in elongation by 9%.

4. Conclusion

Treatment of two low-carbon steels by Q&P process was trialled experimentally. Austenitizing temperature and cooling rate were found to play key role in formation of the resulting microstructure.

In the CMnSi, stepwise optimization of processing parameters led to a martensitic microstructure with some amount of ferrite and 12% retained austenite. Even this low-carbon steel, with no other alloying additions but manganese and silicon, showed the strength of 1012 MPa and an elongation of 19%.

In the CCrMnSi steel, which had Cr addition, the strength was higher by 250 MPa at the elongation of 10%. The effect of chromium was mainly reflected in the higher strength and lower elongation level.

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REFERENCES

[1] S. Wiewiorowska, Archives of Metallurgy and Materials **58**, 573 (2013).

[2] J. Speer, D.K. Matlock, B.C. De Cooman, J.G. Schroth, Acta Materialia **51**, 2611 (2003).

[3] D.V. Edmonds, K. He, B.C. De Cooman, D.K. Matlock, J.G. Speer, Materials Science and Engineering A **438-440**, 25 (2006).

[4] F.L.H. Gerdemann, Microstructure and hardness of 9260 steel heat/treated by the quenching and partitioning process, Diploma Thesis, Aachen University of Technology, Germany, 2004.

[5] E. De Moor, S. Lacroix, A.J. Clarke, J. Penning, J.G. Speer, Metallurgical and Materials Transactions A **39A**, 2586 (2008).

[6] H. Jirková, L. Kučerová, B. Mašek, Materials Science Forum **706-709**, 2734 (2012).

[7] V. Pileček, H. Jirková, B. Mašek, Advanced Materials Research **887-888**, 257 (2014).

[8] H. Jirková, B. Mašek, M.F.-X. Wagner, D. Langmajerová, L. Kučerová, R. Treml, D. Kiener, Journal of Alloys and Compounds (2014), DOI http://dx.doi.org/10.1016/j.jallcom.2013.12.028 (in press).

[9] D. Hauserová, Z. Nový, P. Motyčka, J. Džugan, Journal of Iron and Steel Research International **18 (1-1)**, 417 (2011).

[10] W. Bleck, in: B.C. De Cooman (Ed.), International Conference on TRIP – Aided High Strength Ferrous Alloys, , GRIPS 13-25, Bad Harzburg (2002).

[11] K.W. Andrews, Journal of The Iron and Steel Institute, 721 (1965).

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