UDK 621.77:621.777.2 ISSN 1580-2949
Original scientific article/Izvirni znanstveni članek MTAEC9, 49(5)739(2015)

PRODUCTION OF SHAPED SEMI-PRODUCTS FROM AHS STEELS BY INTERNAL PRESSURE

IZDELAVA POLPROIZVODOV IZ AHS-JEKEL, OBLIKOVANIH Z NOTRANJIM TLAKOM

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Prejem rokopisa – received: 2014-09-04; sprejem za objavo – accepted for publication: 2014-11-05

doi:10.17222/mit.2014.218

The constant evolution of industry brings demands for components of ever-higher quality, complex shapes and excellent mechanical properties, with reduced operating costs and with improved reliability in service. Such demands can be met by processing high-strength, low-alloyed steels using novel heat-treatment methods. One of such methods is the quenching and partitioning (Q&P) process, which can lead to high strengths of about 2000 MPa at elongation levels of more than 10 %. The Q&P process can be combined with unconventional procedures for making complex-shaped parts to manufacture components with excellent mechanical properties and an intricate shape. This paper describes the sequence of internal high-pressure forming, hot stamping and the Q&P process used for making a functional product with good mechanical properties and a complex shape. The purpose of the experimental programme was to employ a production chain for processing hollow, thin-walled stock. Using a step-by-step optimization, commercially usable products were obtained. The microstructure of the products consisted primarily of martensite and a small fraction of bainite. Such a microstructure guarantees a high ultimate strength. The strength level throughout the part was not less than 1950 MPa, combined with an excellent elongation of approximately 15 %.

Keywords: Q&P process, hot stamping, internal high pressure forming

Stalen razvoj industrije zahteva komponente vedno boljših kvalitet, kompleksnih oblik, odličnih mehanskih lastnosti pri nižjih proizvodnih stroških in z večjo zanesljivostjo obratovanja. Take zahteve se lahko dosežejo z izdelavo visokotrdnostnih malolegiranih jekel z uporabo novih metod toplotne obdelave. Ena od takih metod je postopek kaljenja in delitve (Q&P), ki omogoča velike trdnosti okrog 2000 MPa pri več kot 10-odstotnem raztezku. Postopek Q&P se lahko kombinira z nekonvencionalnimi metodami izdelave delov s kompleksno obliko in za izdelavo komponent z odličnimi mehanskimi lastnostmi in komplicirano obliko. Predstavljeni članek obravnava zaporedne operacije notranjega visokotlačnega preoblikovanja, vročega stiskanja in Q&P-postopka za izdelavo funkcionalnih proizvodov z dobrimi mehanskimi lastnostmi in zapleteno obliko. Namen eksperimentalnega dela je bil postaviti proizvodno verigo za izdelavo votlih tankostenskih kosov. Z optimizacijo korak za korakom so bili izdelani komercialno uporabni proizvodi. Mikrostruktura proizvodov je bila sestavljena predvsem iz martenzita in majhnega deleža bainita. Taka mikrostruktura zagotavlja veliko natezno trdnost. Nivo trdnosti po vsem kosu ni bil manjši od 1950 MPa v kombinaciji z odličnim raztezkom okrog 15 %.

Ključne besede: Q&P-proces, vroče stiskanje, preoblikovanje z velikim notranjim tlakom

1 INTRODUCTION

Advanced high-strength steels (AHSS) find an ever-increasing number of uses in a wide range of industrial sectors. Thanks to their excellent mechanical properties, they become the materials of choice for structural parts, which can thus be more lightweight than, but equally reliable and safe as, those made of conventional steels. Due to the continuously evolving needs of industry, new processing methods must always be sought to be combined in a comprehensive production chain.

2 Q&P PROCESSING

The Q&P process is an advanced method of heat treatment for high-strength steels.[1] It relies on the processes that take place in the steel between the M_s and M_f temperatures and are related to carbon diffusion. Carbon migrates from the super-saturated martensite to the retained austenite, making the latter more stable. The Q&P process comprises heating to the austenitizing temperature and quenching to the quenching temperature (QT), which lies between the M_s and M_f temperatures. This produces a mixture of martensite and retained austenite.[2,3] The workpiece is then heated to, and held at, the partitioning temperature (PT).[4]

With regard to the austenite stabilization, carbon must not precipitate in the form of carbides at the PT. To ensure this, aluminium, silicon or phosphorus should be used as alloying additions. In order to strengthen the solid solution, the steel can be alloyed with manganese and chromium. These elements shift the pearlitic and bainitic transformation curves in the transformation diagrams towards longer times, thus reducing the critical cooling rate. As silicon is present in the steel in order to retard carbide precipitation, the optimum level of chromium must be controlled, as chromium in the presence

Table 1: Chemical composition of steel 42SiCr, *w*/%
Tabela 1: Kemijska sestava jekla 42SiCr, *w*/%

C	Si	Mn	Cr	Mo	Nb	P	S	M_s/°C	M_f/°C
0.42	2.6	0.59	1.33	0.03	0.03	0.01	0.01	289	178

of carbon acts as a carbide-former. The addition of manganese as an austenite-forming element effectively prevents any free ferrite from forming. Free ferrite can impair the mechanical properties of the steel.[5–7]

2.1 Internal High Pressure Forming

This method can be used for manufacturing complex-shaped, hollow products. It is based on forming a hollow feedstock in a closed die using pressurized gas. The final temperature of the product can be controlled by changing the die-opening time. At the end of this die-opening time, the product is removed from the die and cooled in air or heat treated further.

3 EXPERIMENTAL PROGRAMME

The goal of the experimental programme was to form hollow feedstock using internal, high-pressure gas and then apply the Q&P process. The experimental material was high-strength 42SiCr steel (**Table 1**). The initial microstructure of this steel consisted of ferrite and pearlite. The ultimate strength was 981 MPa, the elongation reached 30 % and the hardness was 295 HV10. Using the JMatPro software, the important M_s and M_f temperatures were found: 289 °C and 178 °C, respectively (**Figure 1**).

3.1 Experimental Production of Shaped Products

The hollow feedstock used for making the shaped products was 380 mm in length and 48 mm in diameter. Its wall thickness was 4 mm (**Figure 2**).

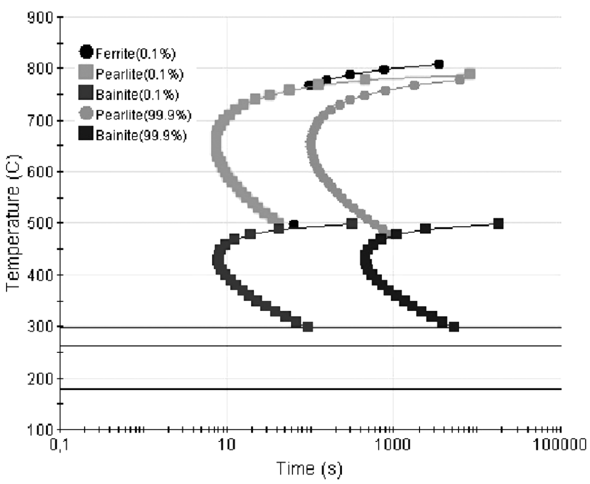

Figure 1: TTT diagram of 42SiCr steel calculated using JMatPro software
Slika 1: TTT-diagram jekla 42SiCr, izračunan s programsko opremo JMatPro

Figure 2: Experimental stock and the resulting shape after hot stamping
Slika 2: Preizkusni kos in dobljena oblika po vročem stiskanju

The sequence comprised heating to 915 °C and holding for 25 min to guarantee full austenitizing. Then the feedstock was transferred to a die at the ambient temperature. After the die was closed, the feedstock was formed by pressurized gas at an overpressure of 700 bar. The working gas was nitrogen. The contact between the workpiece and the die wall caused the workpiece to cool down rapidly to a pre-defined temperature. The die wall was at room temperature and was cooled by water. This was achieved by selecting the appropriate die-opening time between 5 s and 20 s. After the die-opening time, the workpiece is removed. In order to explore the effect of Q&P processing, the final step of the proposed sequence, on the microstructure evolution and the related mechanical properties, the workpieces were further processed in three different ways (**Figure 3**). Some workpieces were cooled in air after hot stamping in the

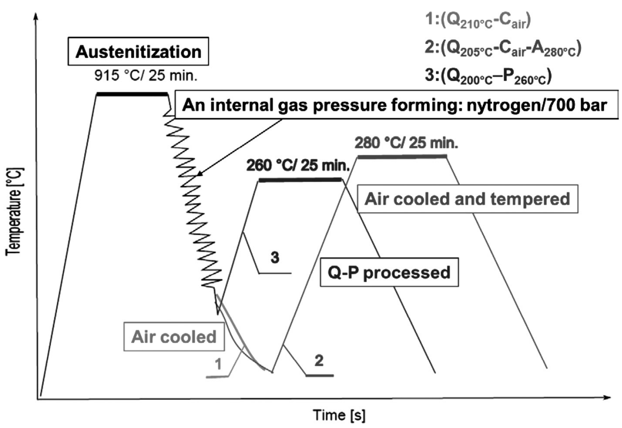

Figure 3: Experimental modes of heat treatment
Slika 3: Eksperimentalni načini toplotne obdelave

die. The product from this group was hot stamped in the die for 15 s and was then removed and cooled in air ($Q_{210\ °C} - C_{air}$). The temperature at the moment of removal from the die was 210 °C. Another group comprised workpieces that were cooled in the die, then cooled in air to the ambient temperature and were then reheated in a furnace to 280 °C, where they were held for 25 min, and then removed and cooled in air. The product from the second group was cooled in the die for 15 s to a temperature of 205 °C ($Q_{150\ °C} - C_{air} - A_{280\ °C}$). Yet another group included workpieces that were removed from the die and immediately placed in a furnace. The significant product from this third group was cooled in the die for 15 s as well. Then it was placed in a furnace and annealed at 260 °C for a time of 25 min ($Q_{200\ °C} - P_{200\ °C}$). The temperature after removal from the die reached 200 °C (**Table 2**). They were therefore treated using the Q&P process.

Table 2: Parameters of the experimental heat treatment
Tabela 2: Parametri eksperimentalne toplotne obdelave

	Austeni-tizing °C	QT-tem-perature °C	PT-tem-perature °C	Temper-ing time min
($Q_{210\ °C} - C_{air}$)	915	210	–	–
($Q_{205\ °C} - C_{air} - A_{280\ °C}$)	915	205	280	25
($Q_{200\ °C} - P_{260\ °C}$)	915	200	260	25

4 RESULTS AND DISCUSSION

Representative products were selected from these groups of workpieces for metallographic examination and for mechanical property measurements. The specimens for the mechanical property measurement were taken from the products representing the different processing sequences. The specimen locations were chosen to allow an evaluation of the effects of the production sequence and of the position within the die upon the resulting product properties. Representative specimens were taken from the parts with which the workpiece was held in the die, from the area of the steepest change in cross-section, which is the point of the largest plastic strain, and from the transitions between the different diameters. A total of 14 test specimens were taken from each product (**Figure 4**)

Due to the shape of the workpieces, miniature tension test specimens with a gauge length of 5 mm and a cross-section of 2 mm × 1.5 mm were used for measuring the mechanical properties (**Figure 5**).

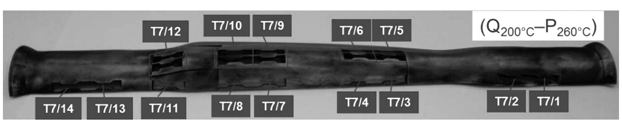

Figure 4: Selected locations of the T7 ($Q_{200\ °C} - P_{260\ °C}$) workpiece, upon the Q&P process
Slika 4: Izbrana področja kosa T7 ($Q_{200\ °C} - P_{260\ °C}$) pri Q&P-procesu

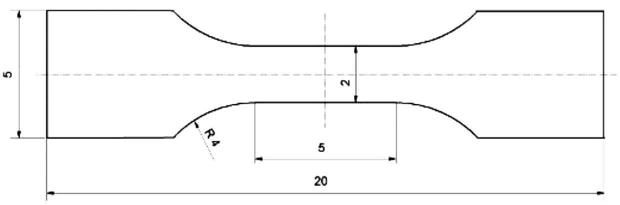

Figure 5: Shape of specimen for mechanical testing
Slika 5: Oblika vzorca za mehanske preizkuse

4.1 Mechanical Properties

The product that was only cooled in air after removal from the die ($Q_{210\ °C} - C_{air}$) showed a very high ultimate strength (**Table 3**). None of the 14 specimens exhibited ultimate strengths of less than 2260 MPa. The elongation level was higher than 10 % in all cases. The specimen with the lowest ultimate strength, 2263 MPa, and an elongation of 12 %, was T3/5, which had been taken from the transition to the largest diameter. It also showed the lowest hardness of all the specimens: 667 HV10. It is likely that this part of the workpiece was not in full contact with the die wall. No similar effect of the specimen position was found in any other specimen from this product.

Table 3: Results of tensile testing (proof stress $R_{p0.2}$, ultimate tensile strength R_m and elongation $A_{5\ mm}$) and hardness testing (HV10) of the semi-product ($Q_{210\ °C} - C_{air}$) upon air cooling
Tabela 3: Rezultati nateznih preizkusov (meja tečenja $R_{p0,2}$, natezna trdnost R_m in raztezek $A_{5\ mm}$) in merjenja trdote (HV10) polproizvodov ($Q_{210\ °C} - C_{zrak}$) po ohlajanju na zraku

	$R_{p0.2}$/MPa	R_m/MPa	$A_{5\ mm}$/%	HV10
T3/2	1550	2324	14	709
T3/3	1534	2314	11	685
T3/5	1518	2263	12	667
T3/8	1641	2333	10	701
T3/12	1681	2345	10	738
T3/14	1652	2310	16	705

All the specimens from the product ($Q_{205\ °C} - C_{air} - A_{280\ °C}$) exhibited ultimate strengths of more than 2110 MPa and $A_{5\ mm}$ elongation levels of more than 14 % (**Table 4**). A low ultimate strength was found in the T6/5 specimen: 2133 MPa. The T6/5 specimen also showed $A_{5\ mm}$ elongation of 18 %. This situation is similar to that in the product that was removed from the die and cooled

Table 4: Results of tensile testing of the semi-product ($Q_{150\ °C} - C_{air} - A_{280\ °C}$)
Tabela 4: Rezultati nateznih preizkusov polproizvodov ($Q_{150\ °C} - C_{zrak} - A_{280\ °C}$)

	$R_{p0.2}$/MPa	R_m/MPa	$A_{5\ mm}$/%	HV10
T6/2	1822	2117	18	656
T6/3	1862	2160	17	653
T6/5	1838	2133	18	672
T6/8	1886	2189	18	637
T6/12	1771	2144	14	623
T6/14	1818	2196	19	671

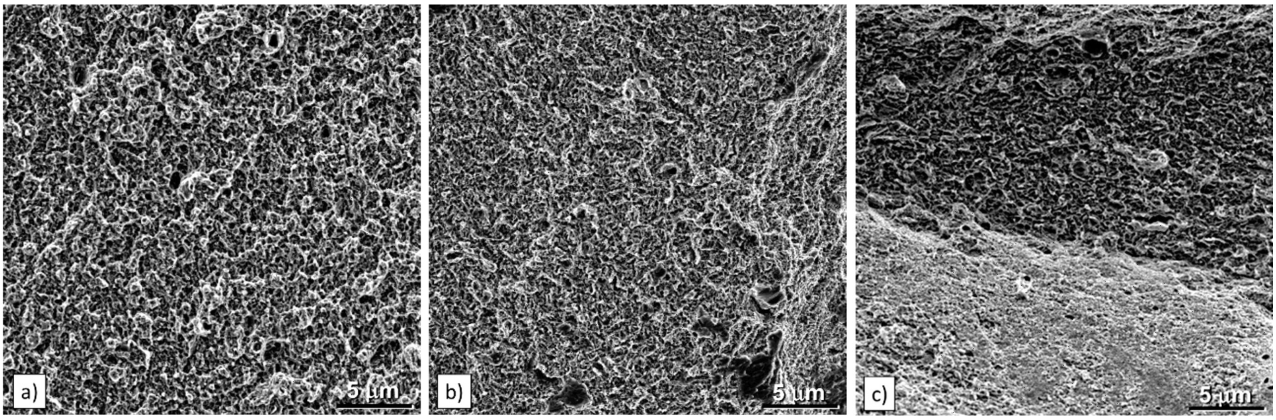

Figure 6: Fracture surfaces: a) T3 ($Q_{200\ °C} – C_{air}$) product that was cooled in air (T3/3), b) T6 ($Q_{205\ °C} – C_{air} – A_{280\ °C}$) that was cooled in air and then annealed in a furnace (T6/3), c) T7 ($Q_{200\ °C} – P_{260\ °C}$) product after Q&P process (T7/3)

Slika 6: Površine prelomov: a) T3 ($Q_{200\ °C} – C_{zrak}$)-proizvod, ohlajen na zraku (T3/3), b) T6 ($Q_{205\ °C} – C_{zrak} – A_{280\ °C}$), ohlajen na zraku in potem žarjen v peči (T6/3), c) T7 ($Q_{200\ °C} – P_{260\ °C}$) proizvod po Q&P-postopku (T7/3)

in air. By contrast, the hardness in the corresponding location of the latter product was the highest of all the locations tested: 672 HV10.

The ultimate strength of the product ($Q_{200\ °C} – P_{260\ °C}$) was approximately 250 MPa to 300 MPa lower than that of the product that had been removed from the die and merely cooled in air (**Table 5**). In addition, it is approximately 150 MPa to 200 MPa lower than in the product that was removed from the die, cooled in air and then reheated to 280 °C and held for 25 min. As in the previous cases, the lowest ultimate strength was found in the T7/5 location. It was 1914 MPa. The corresponding $A_{5\ mm}$ elongation was 20 % and the hardness reached 600 HV10. The specimens in the remaining locations exhibited strengths of approximately 2000 MPa. The Q&P processing of the product improved its elongation. It was between 17 % and 21 %.

Table 5: Results of tensile testing of the product ($Q_{200\ °C} – P_{260\ °C}$) prepared using the Q&P process

Tabela 5: Rezultati nateznih preizkusov proizvodov ($Q_{200\ °C} – P_{260\ °C}$), izdelanih po Q&P-postopku

	$R_{p0.2}$/MPa	R_m/MPa	$A_{5\ mm}$/%	HV10
T7/2	1576	1978	17	623
T7/3	1490	1956	21	615
T7/5	1379	1914	20	600
T7/8	1600	2006	18	596
T7/12	1584	1952	18	597
T7/14	1626	1978	17	608

4.2 Metallographic and Fractographic Characterization

With reference to the results of the tension test, the fracture surfaces were examined. In all cases, ductile fractures with dimples were found (**Figure 6**). No signs of brittle fracture were detected, even in the product that was cooled in air after quenching in the die.

The microstructure was examined in locations corresponding to those of the samples taken for mechanical testing. In almost all the specimens taken from the product ($Q_{200\ °C} – C_{air}$) the microstructure consisted of martensite and a small proportion of bainite (**Figure 7**). Only those areas where the workpiece was not in full

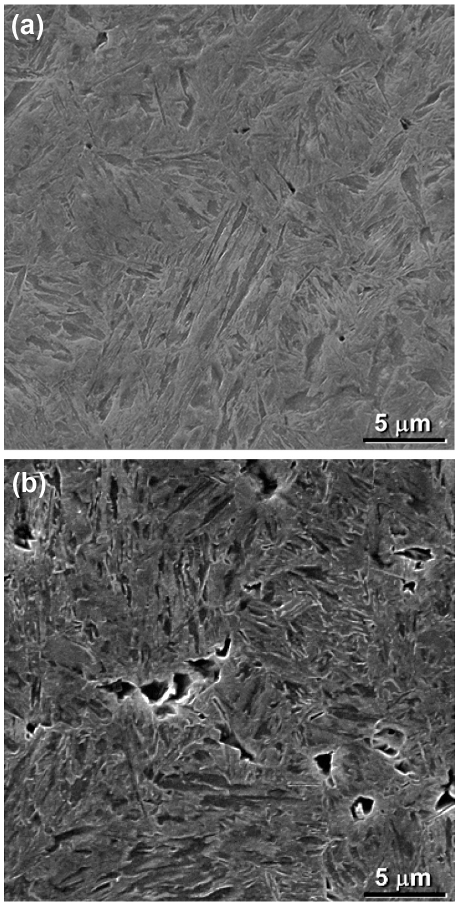

Figure 7: T3 ($Q_{200\ °C} – C_{air}$) product upon air cooling: a) martensite-bainite microstructure (point T3/2), b) martesite-bainite with a small fraction of ferrite (point T3/12)

Slika 7: T3 ($Q_{200\ °C} – C_{zrak}$)-proizvod po ohlajanju na zraku: a) martenzitno-bainitna mikrostruktura (točka T3/2), b) martenzit-bainit z majhnim deležem ferita (točka T3/12)

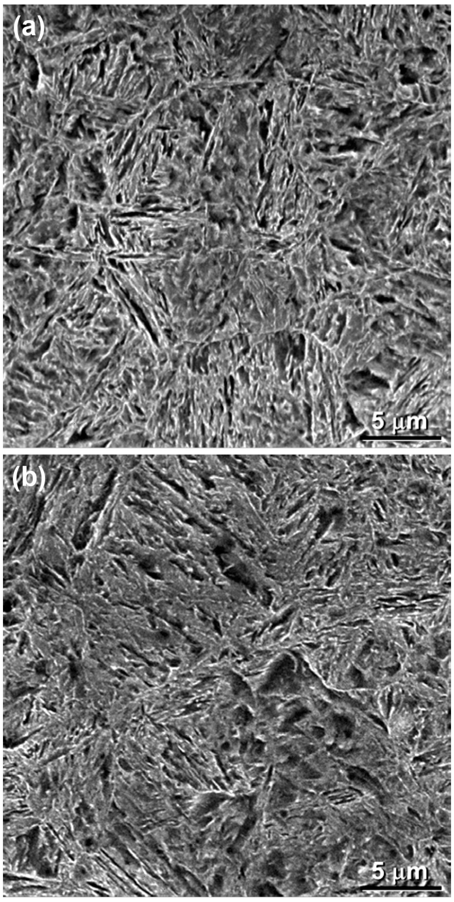

Figure 8: T6 ($Q_{205\ °C}$ – C_{air} – $A_{280\ °C}$) product upon air cooling and annealing: a) tempered martensite-bainite microstructure (point T6/2), b) tempered martensite-bainite microstructure (point T6/12)

Slika 8: T6 ($Q_{205\ °C}$ – C_{zrak} – $A_{280\ °C}$)-proizvod po ohlajanju na zraku in popuščanju: mikrostruktura: a) popuščeni martenzit-bainit (točka T6/2), b) mikrostruktura: popuščeni martenzit-bainit (točka T6/12)

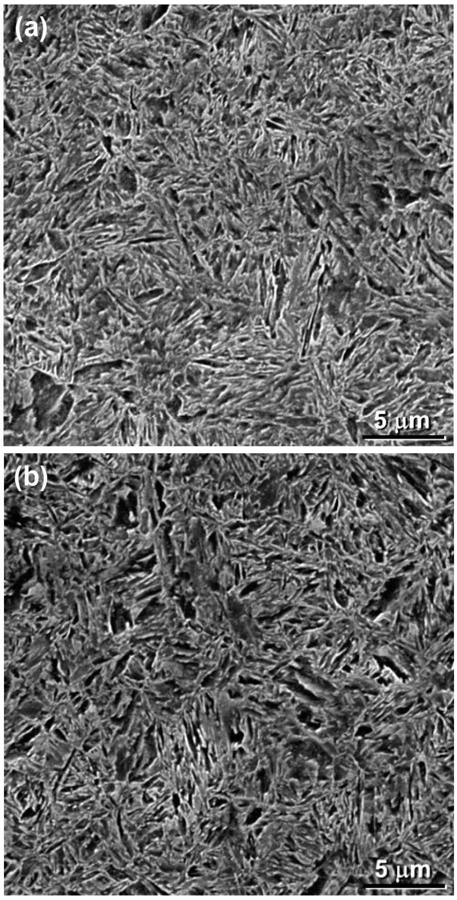

Figure 9: T7 ($Q_{200\ °C}$ – $P_{260\ °C}$) product upon Q&P process: a) martensite-bainite microstructure with a small fraction of ferrite (point T7/2), b) martensite-bainite with a small fraction of ferrite (point T7/12)

Slika 9: T7 ($Q_{200\ °C}$ – $P_{260\ °C}$) proizvod po Q&P postopku: a) martenzitno-bainitna mikrostruktura z majhnim deležem ferita (točka T7/2), b) martenzit-bainit z majhnim deležem ferita (točka T7/12)

contact with the die wall contain some free ferrite, which is probably due to the resulting insufficient cooling rate

An examination of the microstructure of the product ($Q_{205\ °C}$ – C_{air} – $A_{280\ °C}$) revealed tempered martensite and bainite (**Figure 8**). The locations where the workpiece was not in full contact with the die wall contained a small amount of free ferrite.

The Q&P-processed product ($Q_{200\ °C}$ – $P_{260\ °C}$) contained a mixture of martensite and bainite (**Figure 9**). As in the products mentioned above, the locations of partial contact between the workpiece and the die wall contained a small amount of free ferrite.

5 CONCLUSION

Using internal high-pressure forming, products with complex shapes were obtained. A detailed inspection of their surfaces revealed no distinct discontinuities. In the product that was cooled in air after removal from the die, the 15 s in the die caused quenching to 210 °C. Its microstructure was a mixture of martensite, bainite and a small proportion of free ferrite. No location of the product showed an ultimate strength of less than 2260 MPa. The elongation was more than 10 %.

The product that was cooled in air after removal from the die and then annealed in a furnace had a temperature of 205 °C after the 15 s die-opening time. The annealing of the product at 280 °C for 25 min produced a microstructure of tempered martensite, bainite and a small amount of free ferrite. No location of the product, where the mechanical properties were measured, showed an ultimate strength of less than 2110 MPa. Annealing after removal of the product from the die and subsequent cooling in air caused the elongation level to increase from 10 % to 14 %.

Cooling in the die for 15 s, which is used as part of the Q&P process, led to the quenching of the product to 200 °C. The subsequent reheating to the carbon-partitioning temperature of 260 °C and holding for 25 min resulted in a microstructure that consisted of tempered martensite, bainite and free ferrite. The ultimate strength of the product did not decrease below 1900 MPa at any location. The Q&P processing led to an increase in the elongation from 10 % to 18 %.

The measurement of mechanical properties by means of tension testing did not identify any substantial effect of the location, and thus no substantial effect of the strain magnitude on the ultimate strength.

Acknowledgment

This paper includes results achieved in the project CZ.1.05/3.1.00/14.0297 Technological Verification of R&D Results II, individual activity Hollow Shafts for Passenger Cars Produced by Heat Treatment with the Integration of Q-P Process and the project GAČR P107/12/P960 Influence of a Structure Modification on Mechanical Properties of AHS Steel. The paper also includes results from the project SGS-2013-028 Support of Students Research Activities in Materials Engineering Field.

6 REFERENCES

[1] L. Kucerova et al., Comparison of Microstructures and Properties Obtained After Different Heat Treatment Strategies of High Strength Low Alloyed Steel, Journal of Iron and Steel Research International, 18 (**2011**), 427–431

[2] J. Speer et al., Carbon Partitioning into Austenite after Martensite Transformation, Acta Materialia, 51 (**2003**), 2611–2622, doi:10.1016/S1359-6454(03)00059-4

[3] J. Speer et al., The Quenching and Partitioning Process: Background and Recent Progress, Materials Research, 8 (**2005**) 4, 417–423, doi:10.1590/S1516-14392005000400010

[4] T. Tsuchiyma et al., Quenching and partitioning treatment of a low-carbon martensitic stainless steel, Materials Science and Engineering A, 532 (**2012**), 585–592, doi:10.1016/j.msea.2011.10.125

[5] H. Jirkova, L. Kucerova, B. Masek, Effect of Quenching and Partitioning Temperatures in the Q-P Process on the Properties of AHSS with Various Amounts of Manganese and Silicon, Materials Science Forum, 706–709 (**2012**), 2734–2739, doi:10.4028/www.scientific.net/MSF.706-709.2734

[6] L. Kucerova et al., Optimization of Q-P Process Parameters with Regard to Final Microstructures and Properties, Annals of Daaam For 2009 & Proceedings of the 20th International Daaam Symposium, 20 (**2009**), 1035–1036

[7] B. Masek et al., Improvement of Mechanical Properties of automotive Components Using Hot Stamping with Integrated Q-P Process, Journal of Iron and Steel Research International, 18 (**2011**) 1–2, 730–734