## MECHANICAL PROPERTIES OF A HIGH Si AND Mn STEEL HEAT TREATED BY TWO-STEP QUENCHING AND PARTITIONING

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Steels with high strength and good ductility can be produced using the quenching and partitioning (Q&P) process. These materials can be used as the third generation of advanced high-strength steels in automotive industry due to their high toughness and energy absorption capacity. Due to its high importance, the present study was focused on the evaluation of mechanical properties of a high Si and Mn low-alloy steel during the application of two-step Q&P process. For this aim, the cycles with different partitioning times from 10 to 1000 s were applied and the mechanical properties in terms of strength and elongation were measured and discussed. The final results of this work showed that the considerable UTS, elongation and products of strength and elongation such as 850 MPa, 15.46% and 11.45 GPa% can be provided in samples treated via the Q&P process.

Keywords: Two-step quenching and partitioning, partitioning time, mechanical properties.

Advanced high-strength steels containing substantial amounts of retained austenite have been the subject of many recent investigations due to their good combination of strength and ductility [1-4]. The quenching and partitioning (Q&P) process proposed by Speer et al. [5, 6] is a promising heat treatment to manufacture advanced highstrength steels. The process consists of the first quench, from either full or partial austenitization to a temperature between the martensite start temperature  $(M_s)$  and the martensite finish temperature  $(M_f)$ , a subsequent isothermal treatment at the same or higher temperature for the partitioning of carbon from the supersaturated martensite  $(\alpha')$  to the remaining austenite  $(\gamma)$  and a final quench to the room temperature, at which sufficiently stable austenite is retained. The Q&P process leads to microstructures containing martensite and carbon-enriched retained austenite where martensite acts as a strengthening phase while retained austenite contributes significantly to the ductility. Two effects of retained austenite on ductility have been proposed. One is the transformation-induced plasticity (TRIP) effect, which involves relaxing local stress concentration through austenite to martensite transformation [7]. The other is the blocking microcrack propagation (BMP) effect [8]. However, other possible effects of retained austenite on ductility during uniform deformation have not yet been investigated. Influence of the temperature of quenching/partitioning on the morphology of 37MnSi5 steel was studied in previous literature [9]. In this investigation, two-step Q&P process cycles with different partitioning times from 10 to 1000 s were applied to a high Si and Mn low-alloy steel and mechanical properties in terms of strength and elongation were evaluated.

**Experimental procedures.** The base material used was a low alloy C–Mn–Si steel with a composition similar to conventional TRIP-assisted steels (wt.%): 0.362 C; 1.38 Si; 1.24 Mn; 0.0973 Cr; 0.0902 Ni; 0.0711 Cu; 0.03 Al; 0.025 Pb; 0.0245 P; 0.0202 S; 0.015 W; 0.0101 Co; 0.0101 As; 0.0095 Sn; 0.005 Mo; 0.0025 Nb; 0.0023 Ti; 0.002 B; 0.002 Zr; 0.002 V. The critical temperatures for the base material were  $M_s = 339^{\circ}$ C,

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 $Ac_1 = 748.1$ °C and  $Ac_3 = 841.5$ °C, respectively, which were obtained by dilatometric analysis.

Tensile test samples were prepared according to ASTM E8-04 with a gauge length of 25.4 mm and the X-ray diffraction (XRD) samples were  $15 \times 10 \times 3$  mm. In this investigation, 3 tensile samples and 3 XRD samples were used for each condition of the test and the average of obtained results was reported. For the two-step Q&P heat treatment, the samples were heated to 900°C at a heating rate of  $+5^{\circ}$ C/s in a furnace and held for 10 min for full austenitization, then quenched into an oil bath at 238°C (optimum quenching temperature) with a cooling rate of 220°C/s. They were heat treated at 400°C in a molten salt bath for 10; 30; 100; 400; 700 and 1000 s, respectively. Finally, they were water quenched to room temperature.

After the heat treatments, the yield strength, ultimate tensile strength (UTS) and elongation were measured at room temperature at an extension rate of 0.5 mm/min using a GOTECH MACHINE-1220AJ-50K testing machine.

In order to determine the mean crystallite size and dislocation density in the final phases of the treated specimens, XRD measurements were performed on a Bruker D8 diffractometer using  $CuK_{\alpha}$ -radiation operating at 35 kV and 30 mA. Samples were scanned over a 2 $\theta$  range from 10 to 90 degrees with a dwell time of 1 s and a step size of 0.05 degree. The mean crystallite size of the retained austenite and martensite phases was determined using Scherrer's Eq. (1) [10] on the  $(200)_{\gamma}$ ,  $(220)_{\gamma}$  and  $(200)_{M}$ ,  $(211)_{M}$  peaks, and the dislocation density of martensite phase was calculated using Eq. (2) [11, 12]:

$$D = \frac{K\lambda}{\beta \cdot \cos\theta},\tag{1}$$

where *D* is the mean crystallite size; *K* is Scherrer's constant (shape factor);  $\lambda$  is the X-ray wavelength;  $\beta$  is the width of the XRD peak at a half height;  $\theta$  is the Bragg angle.

$$\delta = n/D^2, \tag{2}$$

where  $\delta$  is the dislocation density; *n* is constant; *D* is mean crystallite size. *K* was equal to 0.9 [10] and *n* was equal to 1 [11, 12].

**Results.** The chemical composition of the base material should be taken into account in Q&P process. The manganese included in the chemical composition was used to retard ferrite, pearlite, and bainite formation and decrease the bainite start temperature, as well as to enhance the austenite stability. On the other hand, silicon was used to restrict carbide precipitation during the partitioning step [13]. The microstructures obtained in this work are discussed elsewhere [14]. A summary of these results is shown in Table 1 and Fig. 1. Moreover, the mean crystallite sizes and dislocation densities calculated for the different conditions of the two-step Q&P process are shown in Table 2.

 Table 1. Microstructural results obtained for the base material after the two-step Q&P [14]

t <sub>P</sub> , s	Volume fraction of retained austenite, % (error)	Average carbon content of retained austenite, % (error)	Carbide precipitation	Bainite formation
10	9.08 (±0.06)	1.0068 (±0.0040)	No	No
30	12.35 (±0.15)	1.3773 (±0.0055)	Yes	No
100	13.58 (±0.14)	1.3068 (±0.0048)	No	No
400	11.8 (±0.07)	1.6591 (±0.0073)	No	Yes
700	9.08 (±0.09)	1.4773 (±0.0036)	No	Yes
1000	9.55 (±0.12)	1.7068 (±0.0089)	No	Yes



quenched to 238°C, partitioned at 400°C for 10 (*a*); 30 (*b*); 100 (*c*); 400 (*d*); 700 (*e*); to room temperature [14]. (C - carbide; M - martensite; IM - initial martensite; B – bainite; FM – fresh martensite; A - austenite; RA - retained austenite).

10 µm

Table 2. The mean crystallite size and dislocation density of the final phases for different conditions of the two-step Q&P

$t_P$ , s	$D_{ m M}$	$D_{\gamma}$	$\delta_{\rm M} \cdot 10^{-10}, \ {\rm cm}^{-2}$	
<i>1p</i> , 5	nm		$\mathrm{cm}^{-2}$	
10	25.63	17.85	15.22	
30	22.18	18.20	20.33	
100	28.88	17.41	11.99	
400	21.66	79.81	21.31	
700	24.03	31.42	17.32	
1000	18.34	20.77	29.73	

The mechanical properties are plotted in Figs. 2 and 3. The UTS and elongation obtained were from 712 to 850 MPa and from 11.22 to 15.46% for these values of partitioning time. Since the strength of the retained austenite is very small, and the volume fraction of this phase is not more than 14 vol.% in this research, the strength and volume fraction of martensite phase are two dominant parameters in determining the final strength of the treated samples. The strength of the martensite phase is derived from various strengthening mechanisms such as: dislocation strengthening of the martensite; precipitation strengthening effect from the carbide particles within martensite; high degree of carbon supersaturation in virgin martensite. According to Fig. 2, the highest UTS and yield strength were 850 and 672 MPa, which were obtained for the shortest partitioning time (10 s), which could be due to a maximum volume fraction of martensite and a maximum degree of carbon supersaturation in virgin martensite in this condition. Yield strength and UTS decreased to 621 and 802 MPa, respectively with increasing of partitioning time from 10 to 30 s. Despite the decreasing of strength, the yield strength level in this condition was higher than that of the longer partitioning times, which was due to the carbide particles precipitating during partitioning which could provide a considerable precipitation strengthening effect. A minimum volume fraction of the martensite phase and minimum dislocation density in this phase  $(11.99 \cdot 10^{10} \text{ cm}^{-2})$ resulted in decreased strength in the sample partitioned for 100 s. Increase of the volume fraction of martensite and the dislocation density to  $21.31 \cdot 10^{10}$  cm<sup>-2</sup>, increased the mechanical strength for the partitioning time of 400 s. Decreasing in volume fraction of retained austenite and increasing the average carbon content of this phase encouraged bainite formation during partitioning between 400 and 1000 s. Banitie formation increases strength. Moreover, the maximum dislocation density in the martensite phase for partitioning time of 1000 s (29.73  $10^{10}$  cm<sup>-2</sup>) helped to increase the strength level for this condition.



Fig. 2. Effect of partitioning time on strength of samples treated by the two-step Q&P:  $\blacksquare$  – yield;  $\blacktriangle$  – UTS.

Fig. 3. Effect of partitioning time on elongation of samples treated by the two-step Q&P  $(\square 2 - step)$ .

Retained austenite plays a key role in plasticity enhancement. Interlath film-like austenite can impede the generation and propagation of cracks and in turn improve toughness effectively [15]. Furthermore, both interlath and island-like austenite can partially transform to martensite and show the "TRIP" effect during deformation, eliminating stress concentration and retarding the occurrence of necking [16], which results in increased elongation. With increased retained austenite volume fraction, more martensitic transformation from the TRIP phenomenon during deformation occurred, and led to increased elongation and formability of steel sheet.

According to Fig. 3, the greatest elongation in this research was 15.46% which was obtained for the sample partitioned for 100 s. It was due to the maximum volume fraction of retained austenite under this condition (13.58%), the thicker austenitic films



Fig. 4. Products of tensile strength and total elongation of Q&P processed base material  $(\blacksquare 2 - \text{step}).$ 

and many austenitic islands with sizes much larger than a micron. Also, the minimum strength in this condition helped to reach the maximum ductility. An elongation of 14.28% was reached for partitioning time of 30 s with 12.35% volume fraction of retained austenite. The minimum elongation in this research was 11.22% which was obtained for the partitioning time of 10 s, and was due to a minimum

volume fraction of retained austenite of 9.08%. Also, the maximum strength in this condition helped to reach a minimum ductility. Nevertheless, a smaller volume fraction of retained austenite at the partitioning time of 700 s compared to 1000 s, offered a higher ductility. The formation of bainite in partitioning times from 400 to 1000 s decreased the volume fraction of retained austenite. Also, bainitic ferrite can act as a barrier against the autocatalytic propagation of the austenite to martensite transformation [17], so it can decrease the elongation for partitioning times longer than 100 s.

The product of strength and elongation indicates the balance of strength and ductility for advanced high-strength steels in engineering applications [18]. The products of tensile strength and total elongation (UTS×El.) of the Q&P processed base material were calculated and are given in Fig. 4. The UTS×El. of Q&P processed base material is from 9.12 to 11.45 GPa% and these values are considerable compared with similar heat treatments such as austempering (~ 10 GPa%) [19] and conventional quenching and tempering (~ 6 GPa%) [19] for industrial applications.

## CONCLUSIONS

In this research, Q&P heat treatments, where the partitioning step was performed at a higher temperature than the quenching temperature, were applied to a high Si and Mn steel. The products of tensile strength and total elongation of Q&P processed base material in this research were from 9.12 to 11.45 GPa% and this range is considerable comparing with similar heat treatments such as austempering ( $\sim 10$  GPa%) and conventional quenching and tempering ( $\sim 6$  GPa%) for industrial applications. Retained austenite plays a key role in plasticity enhancement. The interlath film-like austenite can impede generation and propagation of cracks and in turn improve toughness effectively. Furthermore, both interlath and island-like austenite can partially transform to martensite and show "TRIP" effect during deformation, eliminating stress concentration and retarding the happening of necking, which results in the increasing of elongation.

*РЕЗЮМЕ*. Високоміцні сталі з підвищеною в'язкістю можна виготовляти гартуванням та фрагментуванням (Q&P). Їх можна вважати третім поколінням матеріалів для використання в автомобільній промисловості. Акцентовано увагу на оцінці механічних властивостей низьколегованої сталі з високим вмістом Si та Mn під час двоступінчастого процесу Q&P. Для цього використано цикли з різним часом фрагментування (від 10 до 1000 s), а також вивчено та проаналізовано міцність та відносне видовження. Виявлено, що високі значення границі міцності, відносного видовження та добутку міцності і видовження, які становлять відповідно 850 MPa, 15,46% і 11,45 GPa%, можна отримати на зразках, оброблених Q&P.

*РЕЗЮМЕ*. Высокопрочные стали с повышенной вязкостью можно изготавливать закалкой и фрагментированием (Q&P). Их можно считать третьим поколением материалов для использования в автомобильной промышленности. Акцентировано внимание на оценке механических свойств низколегированной стали с высоким содержанием Si и Mn при двухступенчатом Q&P. Для этого использованы циклы с разным временем фрагментирования (от 10 до 1000 s), а также определены и проанализированы прочность и относи-

тельное удлинение. Выявлено, что высокие значения предела прочности, относительного удлинения и произведения прочности и удлинение, которые составляют соответственно 850 MPa, 15,46% и 11,45 GPa%, можно получить на образцах, обработанных Q&P.

- The developments of cold-rolled TRIP-assisted multiphase steels. Al-alloyed TRIP-assisted multiphase steels / P. J. Jacques, E. Girault, A. Mertens, B. Verlinden, J. Van Humbeeck, and F. Delannay // ISIJ Int. – 2001. – 41. – 1068 p.
- Very strong bainite / F. G. Caballero and H. K. D. H. Bhadeshia // Curr. Opin. Solid State Mater. Sci. – 2004. – 8. – 251 p.
- Partitioning of carbon from supersaturated plates of ferrite, with application to steel processing and fundamentals of the bainite transformation / J. G. Speer, D. V. Edmonds, F. C. Rizzo, and D. K. Matlock // Curr. Opin. Solid State Mater. Sci. 2004. 8. 219 p.
- Theoretical design and advanced microstructure in super high strength steels / F. G. Caballero, M. J. Santofimia, C. Garcia–Mateo, J. Chao, and C. G. de Andres // Mater. Des. 2009. 30. 2077 p.
- Austenite formation and decomposition / J. G. Speer, A. M. Streicher, D. K. Matlock, F. C. Rizzo, and G. Krauss // Eds.: E. B. Damm, M. Merwin. – Warrendale, PA: TMS/ISS. – 2003. – 505 p.
- The "Quenching and Partitioning" Process: Background and Recent Progress / J. G. Speer, F. C. Rizzo, D. K. Matlock, and D. V. Edmonds // Mater. Res. – 2005. – 8. – 417 p.
- TRIP-assisted steels: cracking of high-carbon martensite / S. Chatterjee, H. K. D. H. Bhadeshia // Mater. Sci. Technol. – 2006. – 22. – 645 p.
- Effects of volume fraction and stability of retained austenite on formability in a 0.1C-1.5Si-1.5Mn-0.5Cu TRIP-aided cold-rolled steel sheet / C. G. Lee, S. J. Kim, T. H. Lee, and S. Lee // Mater. Sci. Eng. A. - 2004. - 371. - 16 p.
- Influence of the temperature of quenching/partitioning on the morphology of 37MnSi5 steel / H. R. Ghazvinloo and A. Honarbakhsh-Raouf // Materials Science. – 2017. – 52, № 4. – P. 572–579.
- Cullity B. D. Elements of X-ray Diffraction, 2<sup>nd</sup> ed. Addison-Wesley Publishing Co. Inc., Boston, 1978.
- Influence of thermal annealing on the composition and structural parameters of DC magnetron sputtered titanium dioxide thin films / B. Karunagaran, R. T. R. Kumar, D. Mangalaraj, S. K. Narayandass, and G. M. Rao // Cryst. Res. Technol. – 2002. – 37. – 1285 p.
- The effect of substrate temperature on growth of nanosilver layer deposited on white glass by magnetron sputtering / N. RahmaniNasab, S. Baghshahi, M. A. Shahbazi, and M. Tamizifar // J. Color Sci. Technol. – 2008. – 2. – 41 p.
- Microstructural development during the quenching and partitioning process in a newly designed low-carbon steel / M. J. Santofimia, L. Zhao, R. Petrov, C. Kwakernaak, W. G. Sloof, and J. Sietsma // Acta Mater. – 2011. – 59. – 6059 p.
- Effect of partitioning time on microstructural evolution of a C–Mn–Si steel in two-step quenching and partitioning process / H. R. Ghazvinloo and A. Honarbakhsh-Raouf // J. Mater. Environ. Sci. – 2014. – 5. – 1988 p.
- Structure-property relations and the design of Fe–4Cr–C base structural steels for high strength and toughness / B. V. N. Rao and G. Thomas // Metall. Trans. A. – 1980. – 11. – 441 p.
- Mechanical and transformation behaviors of a C–Mn–Si–Al–Cr TRIP steel under stress / X. D. Wang, B. X. Huang, Y. H. Rong, and L. Wang // J. Mater. Sci. Technol. – 2006. – 22. – 625 p.
- Trip and its kinetic aspects in austempered 0.4C–1.5Si–0.8Mn steel / O. Matsumura, Y. Sakuma, and H. Takechi // Scripta Metall. – 1987. – 21. – 1301 p.
- High strength-elongation product of Nb-microalloyed low-carbon steel by a novel quenching– partitioning–tempering process / S. Zhou, K. Zhang, Y. Wang, J. F. Gu, and Y. H. Rong // Mater. Sci. Eng. A. – 2011. – 528. – 8006 p.
- Ghazvinloo H. R. Study on phase and microstructuretransformation in quenching and partitioning process of a C-Mn-Si low alloy steel, Ph.D. Thesis in Materials Science. – Semnan University, 2015.

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