# EFFECTS OF COOLING RATE ON THE VOLUME FRACTION OF RETAINED AUSTENITE IN FORGINGS FROM HIGH-STRENGTH MN-SI STEELS

Dagmar BUBLÍKOVÁ<sup>1)</sup>, Hana JIRKOVÁ<sup>1)</sup>, Kateřina RUBEŠOVÁ<sup>1)</sup>, Michal PEKOVIĆ<sup>1)</sup>, Julie VOLKMANNOVÁ<sup>1)</sup>, Marcel GRAF<sup>2)</sup>

<sup>1)</sup>University of West Bohemia, RTI - Regional Technological Institute, Univerzitní 22, CZ – 306 14 Pilsen, Czech Republic

<sup>2)</sup>TU Chemnitz, Professorship Virtual Production Engineering, Straβe der Nationen 62, 09111 Chemnitz, Germany

Corresponding author: e-mail: <u>dagmar.bublikova@seznam.cz</u>, Tel.: +420 720 401 659, Laboratory of Experimental Forming, Faculty of Mechanical Engineering, University of West Bohemia, RTI - Regional Technological Institute, Univerzitní 22, CZ – 306 14 Pilsen, Czech Republic

# Abstract

Various ways are sought today to increase mechanical properties of steels while maintaining their good strength and ductility. Besides effective alloying strategies, one method involves preserving a certain amount of retained austenite in a martensitic matrix. The steel which was chosen as an experimental material for this investigation contained 2.5% manganese, 2.09% silicon and 1.34% chromium, with additions of nickel and molybdenum. An actual closed-die forged part was made of this steel. This forged part was fitted with thermocouples attached to its surface and placed in its interior and then treated using the Q&P process. Q&P process is characterized by rapid cooling from a soaking temperature to a quenching temperature, which is between the Ms and the Mf, and subsequent reheating to and holding at a partitioning temperature where retained austenite becomes stable. The quenchant was hot water. Cooling took place in a furnace. Heat treatment profiles were constructed from the thermocouple data and the process was then replicated in a thermomechanical simulator. The specimens obtained in

this manner were examined using metallographic techniques. The effects of cooling rate on mechanical properties and the amount of retained austenite were assessed. The resultant ultimate strength was around 2100 MPa. Elongation and the amount of retained austenite were 15% and 17%, respectively. Microstructures and mechanical properties of the specimens were then compared to the real-world forged part in order to establish whether physical simulation could be employed for laboratory-based optimization of heat treatment of forgings.

Keywords: closed-die forgings, Q&P process, retained austenite, thermomechanical simulator

### 1 Introduction

One of the current trends, particularly in the forging industry, is to achieve good mechanical properties and thus long life in products at minimized costs. The available heat treatment methods which can impart high strength and ductility to a material include the Q&P process (Quenching and Partitioning), which leads to strengths in excess of 2000 MPa and elongation levels of about 10% [1-5]. It is characterized by rapid cooling from the austenite region to a temperature between the Ms and Mf temperatures, where martensite forms, whereas some austenite remains untransformed. During subsequent isothermal holding, retained austenite (RA) becomes stabilised thanks to carbon which migrates from super-saturated martensite to austenite. According to current knowledge, this retained austenite exists primarily in the form of thin foils between martensite laths or plates [6-8]. To ensure that retained austenite becomes stable, it is important to use the right cooling rate and alloying strategy. They should provide the stability of retained austenite, prevent carbide precipitation within martensite and depress the Ms and Mf [9]. In a majority of advanced high-strength steels, the Ms temperature is in the range of  $350^{\circ}C-$ 400°C. The steel that was used in these experiments was specially designed, with manganese as the dominant alloy addition. In steels of this kind, manganese at higher levels depresses the  $M_s$ and  $M_{\rm f}$  temperatures, and therefore enables quenchants other than salt baths to be used. This reduces costs, and is therefore significant to the economy of the process.

# 2 Experiments

A special composition was designed for a 0.42 % carbon steel to depress the Ms and Mf, using iterative optimization in the JMatPro program. The Mf was below 100°C, thanks to which boiling water could be used for quenching (**Tab. 1**). The reduction in Ms and Mf was due mainly to a higher manganese level, i.e. 2.5 % [10]. Other alloying elements included silicon, chromium, and molybdenum [11]. The purpose of silicon was to prevent carbides from forming, to facilitate the supersaturation of martensite with carbon and to provide solid solution strengthening. Chromium improves hardenabil-ity and strengthens the solid solution. Molybdenum was added with a view to depress the Ms and Mf and improve the stability of austenite. Nickel was added in a small amount. It makes austenite more stable during cooling, improves hardenability and provides solid solution strengthening. Niobium belongs to the most common microalloying elements. Even a minute amount substantially alters mechanical properties of steel [12, 13]. Niobium has a strong affinity for carbon and nitrogen. It combines with them to form carbonitrides which dictate mechanical properties of the material [14].

#### 2.1 Data acquisition and development of physical simulation regimes

First, a closed-die forged part was made of the experimental steel (**Fig. 1**). The data for developing physical simula-tion regimes to be conducted in a thermomechanical simulator were gathered in the course of heat treatment of the forged part [15-20]. For this purpose, the part was fitted with thermocouples, some attached to its surface (the fastest-cooling part of the forging) and others placed in its interior (the slowest-cooling location). Specifically, one thermocouple was attached to the surface (no. 1) and two thermocouples were placed in the part's interior (no. 2 and 3), (**Fig. 1**). The forged part was then Q&P processed. It was heated in an air furnace at 880°C to a fully-austenitic condition. Since the special alloying of the steel de-pressed the  $M_f$  to 78°C in **Table 1**, it was possible to use boiling water at 100°C as a quenchant. Boiling water

makes a better quenchant than oil or salt baths in terms of safety, the bath quality and degradation, as well as environmental aspects. Once the surface temperature reached approx. 100°C, the part was removed from water and transferred for partitioning for 1 hour in a furnace at 200°C (**Fig. 2**). The thermocouple data from point 1 on the surface indicated that the quenching temperature in that location was 100°C. At points 2 and 3, approx. 10 mm below the surface, the quenching temperature was higher, about 230°C. Several different cooling profiles were thus obtained for several locations across the forged part. The part was then heat-treated again, this time using a different regime. The austenitizing temperature was identical but the cooling step took place in air, until the surface temperature reached 240°C. The purpose was to explore the impact of the cooling rate on the final amount of retained austenite. Austenitizing was followed by partitioning in a furnace at 200°C (**Fig. 3**). In this case, the differences between the measured locations were less distinct than after the water-quenching regime.

Both regimes provided real-world data for developing regimes for a thermomechanical simulator. Chemnitz Univer-sity of Technology, which collaborated on this investigation, carried out numerical modelling of the heat treatment using FE software Simufact.forming 14.0. Processes with several cooling rates were modelled [6]. Under laboratory conditions, the impact of changes in processing parameters, such as cooling rate, quenching tem-perature and partitioning temperature on the microstructure and mechanical properties can be determined.

AHSS	С	Mn	Si	Р	S	Cu	Cr	Ni	Al	Mo	Nb	$\mathbf{M}_{\mathbf{s}}$	$\mathbf{M}_{\mathbf{f}}$
	0.419	2.45	2.09	0.005	0.002	0.06	1.34	0.56	0.005	0.04	0.03	209	78

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

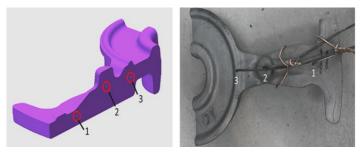


Fig. 1 Closed-die forging of AHSS steel with thermocouples attached for experimental treatment

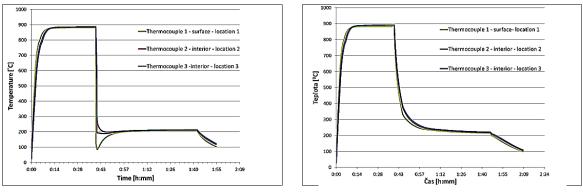


Fig. 2 Q&P processing of the forged part involving quenching in boiling

Fig. 3 Q&P processing of the forged part involving cooling in airprocess

# 2 Physical simulation of cooling of the forged part

Retained austenite in advanced high-strength martensitic steels contributes to their toughness. In order to stabilize retained austenite by Q&P processing, the right quenching temperature must be used along with an appropriate cooling rate. In these experiments, four regimes involving different cooling rates were performed on specimens of the experi-mental steel. The data for designing these regimes were those obtained from heat treatment of the closed-die forged part. The data consisted of cooling curves for quenching in boiling water and for slow cooling in air of the surface and the interior of the forged part (**Fig. 4**).

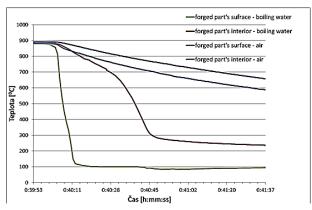


Fig. 4 Detail of cooling curves of the surface and interior of the forged part in the course of quenching in boiling water and cooling in air

The first regime was a simulation of quenching of location 1 on the surface of the forged part in boiling water. In this regime, cooling from the soaking temperature to a quenching temperature

of approximately 100°C took place at the rate of 64°C/s. It was followed by heating to 200°C and holding for 1 hour. In this time interval, retained austenite in the martensitic matrix became stable. The second regime was a physical simulation of quenching of location 3 within the forged part in boiling water. It comprised cooling at 5.7°C/s to the quenching temperature and the same partitioning operation as the previous regime. The third regime was used for simulating air cooling of location 1 on the forged part's surface, using a cooling rate of approx. 3.5°C/s. The subsequent partitioning was performed in a furnace at 200°C for 1 hour. The fourth regime was a physical simulation of air cooling of location 3 in the forged part's interior at a rate of approximately 2.9°C/s (**Fig. 1**). The microstructures and properties of the specimens were then examined and measured using light and scanning electron microscopes and mechanical testing machines, respectively. The amount of retained austenite was determined using X-ray diffraction in **Table 2**.

 Table 2 Heat treatment regimes and mechanical properties

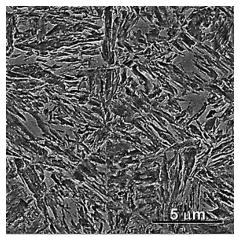
					Physical simulation specimens				Actual forged part				
Regime number	$\begin{array}{c} T_A \\ [^{\circ}C]/t_A[s] \end{array}$	Cooling rate [°C/s]	QT [°C]	PT [°C/s] /t <sub>PT</sub> [s]	HV10 [-]	UTS (R <sub>m</sub> ) [MP a]	A <sub>5mm</sub> [%]	Red. of area [%]	HV10 [-]	UTS (R <sub>m</sub> ) [MPa]	A <sub>5mm</sub> [%]	Red. of area [%]	
1		64	100	200/3600	637	2114	15	17	603	2131	12	10	
2	880/2400	5.7	195	200/3600	669	2250	8	10	643	-	-	9	
3		3.5	240	200/3600	692	2009	3	8	666	1949	2	9	
4		2.9	240	200/3600	690	2141	4	7	656	2000	1	8	

#### 3 Results and discussion

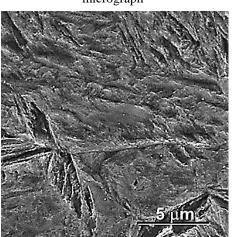
The specimens processed in the thermomechanical simulator were examined using a scanning electron microscope (Tescan Vega 3). All the microstructures consisted of a majority of martensite, a small amount of bainite and various volume fractions of retained austenite (Fig. 5a – 7a). The amount of retained austenite was measured by means of XRD 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). The volume fraction of retained austenite austenite in the martensitic matrix varied with the local cooling rate. Regime 1 led to a strength

of approximately 2100 MPa and elongation of 15% in Table. 2. It was a simulation of quenching of the surface of the forging in boiling water, and therefore it involved fast cooling. A large volume fraction of retained austenite (17%) in the martensitic matrix of this specimen was found by X-ray diffraction analysis. It was confirmed by light microscopic observation (Olympus GX51) of special two-stage-etched metallographic sections (1st etching step: nital, 2nd step: 10% aqueous solution of Na2S2O5) (Fig. 5b). Retained austenite was present as globular grains and as particles between martensite needles. Regime 2 involved slower cooling, at 5.7°C/s. It was a simulation of the forged part's interior during quenching in boiling water. After this regime, the ultimate strength was about 100 MPa higher than in the previous case. As the amount of martensite was larger than in the previous case, hardness was higher by approximately 30 HV10. Elongation dropped substantially to 8%. This can be explained by a lower fraction of RA in Table 2 and by coarser grains. The reduction in the amount of retained austenite (10%) was confirmed by special etching and metallographic observation. There was less RA than after regime 1. Most of it was in the form of globular grains. Some was found between martensitic needles. Two subsequent regimes were similar to each other. They were simulations of air cooling of the surface and interior of the forged part. Their cooling rates were low: 3.5°C/s and 2.9°C/s, respectively. As a consequence, the resulting elongations were even lower than in the previous case – as low as 3%. Related to this was the lower amount of retained austenite: a mere 8%. A larger amount of martensite led to a higher hardness: approx. 690 HV 10 in Table 2, (Fig. 6a). A small amount of retained austenite was in a globular form (Fig. 6b). Mechanical properties of the specimens more or less corresponded to those of the real forged part. The largest amount of retained austenite, 10%, was found in the surface of the forged part upon quenching in boiling water. Re-tained austenite was present as globular grains and as particles between martensite needles (Fig. 7b). The ultimate strength of the forged part, 2131

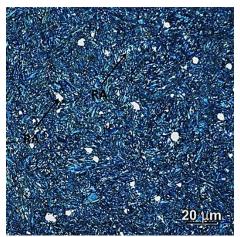
MPa, and its elongation of 12% are nearly identical to those of the physical simulation specimens in **Table 2**.



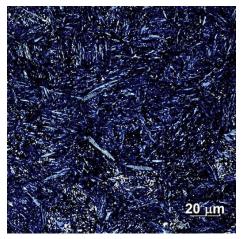
**Fig. 5.** a) Physical simulation of cooling of the forged part's surface, cooling rate: 64°C/s, martensitic structure, bainite, retained austenite, detail scanning electron micrograph



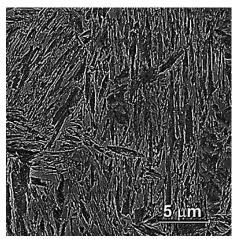
**Fig. 6.** a) Physical simulation of cooling of the forged part's surface, air, cooling rate: 3.5°C/s, martensitic structure, a small amount of bainite, retained austenite, detail scanning electron micrograph



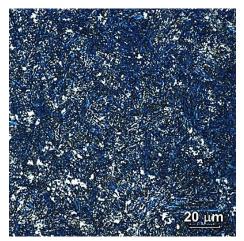
b) Physical simulation of cooling of the forged part's surface, cooling rate: 64°C/s, colour etching to reveal retained austenite, optical micrograph



b) Physical simulation of cooling of the forged part's interior, cooling rate: 3.5°C/s, colour etching to reveal retained austenite, optical micrograph



**Fig. 7.** a) The actual forged part, cooling rate: 64°C/s, boiling water, martensitic structure, bainite, retained austenite, detail scanning electron micrograph



b) The actual forged part, cooling rate: 64°C/s, martensit-ic structure, bainite, retained austenite, colour etching to reveal retained austenite, optical micrograph

## 3 Conclusion

Physical simulation regimes with various cooling profiles (64°C/s, 5.7°C/s, 3.5°C/s and 2.9°C/s) demonstrated the substantial impact that cooling rates have on the resulting amount of retained austenite in martensitic matrix. Upon cooling at the highest rate, which corresponded to quenching of the forged part's surface in hot water, some austenite failed to decompose into martensite. As a result, a considerable volume fraction of austenite remained present as a stable phase in the martensitic matrix, thanks to appropriate alloying. The regime, which involved this particular cooling rate, led to a high elongation, as high as 15%, and an ultimate strength of approximately 2100 MPa. Regimes with slow cooling, which physically simulated cooling of the interior of the forged part in hot water and in air, provided smaller amounts of retained austenite in martensitic matrix, sometimes as low as 8%. Higher hardness levels, which were caused by larger volume fractions of martensite, were associated with markedly lower elongations. Mechanical properties of the AHS steel after physical simulation of treatment of the forged part were in agreement with the values for the actual forged part. Fast cooling (64°C/s) in boiling water led to a strength of 2130 MPa and elongation of 12%.

The comparison between physical simulation and the real-world forged part suggests that physical simulation in the laboratory enables a wide range of heat treatment parameters to be tested for optimizing the processing of closed-die forgings.

### References

- D.V. Edmondsa, K. HEA, F.C. Rizzo, B.C. De Coomanc, D.K. Matlock: Quenching and partitioning martensite - A novel steel heat treatment, Materials Science and Engineering A, 2006, Vol. 438–440, p. 25–34, doi: 10.1016/j.jallcom.2012.02.016
- [2] B. Mašek, H. Jirková, D. Hauserová, L. Kučerová, D. Klaubeová: The Efe Effect of Mn and Si on the Properties of Advanced High Strength Steels Processed by Quenching and Partitioning, Materials Science Forum, Vol. 654-656, 2010, p. 94-97, doi: 10.4028/www.scientific.net/MSF.654-656.94
- [3] H. Jirková, L. Kučerová, B. Mašek: 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, Vol. 706-709, 2012, p. 2734-2739, doi: 10.4028/www.scientific.net/MSF.706-709.2734
- [4] T.Y. Hsu (XuZuyao), X.J. Jin,Y.H.Rong: Strengthening and toughening mechanisms of quenching-partitioning-tempering (Q-P-T) steels, Journal of Alloys and Compounds, Vol. 577, 2013, p. S568–S571, doi: 10.1016/j.jallcom.2012.02.016
- [5] J. G. at al.: The Quenching and partitioning process: Background and Recent Progress. Materials Reasearch. 2005, Vol. 8, č. 4, p. 417-423
- [6] H. Jirková, et al.: Influence of metastable retained austenite on macro and micromechanical properties of steel processed by the Q-P process, Journal of Alloys and Compounds, available online, Journal of Alloys and Compounds, 2014, Vol. 615, p. 163–168, https://doi.org/10.1016/j.jallcom.2013.12.028

- [7] Z. Qian, Q. Lihe, T. Jun, M. Jiangying, Z. Fucheng: Inconsistent effects of mechanical stability of retained austenite on ductility and toughness of transformation-induced plasticity steels, Materials Science & Engineering A, 2013, Vol. 578, p. 370–376, doi.org/10.1016/j.msea.2013.04.096
- [8] E. De Moor, D. Matlock, at al.: Austenite stabilization through manganese enrichment, Scripta Materialia, January 2011, Vol. 64, p. 185–188, https://doi.org/10.1016/j.scriptamat.2010.09.040
- [9] K. Ibrahim, D. Bublíková, H. Jirková, B. Mašek.: Stabilization of Retained Austenite in High-Strength Martensitic Steels with Reduced Ms Temperature, In METAL 2015, Ostrava: TANGER spol. s r. o., 2015, p. 1-7, ISBN: 978-80-87294-58-1
- [10] H. Jirková, L. Kučerová, B. Mašek: 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, Vol. 706-709, 2012, p. 2734-2739, doi: 10.4028 / www.scientific.net / MSF.706-709.2734
- [11]E. V. Pereloma, I. B. Timokina, P.D. Hodgson: Transformation behavior in thermomechanical processed C-Mn-Si steel with and without Nb, Materials Science and Engineering A 273-275 (1999), p. 448-452
- [12] W. Bleck: Using the TRIP efekt the down of a promising group of cod formable steelos, Proccedings of International Conference on TRIP – Aided High Strenght Ferrous Alloys, 2002, Belgium.
- [13] S Baik, S. Kim, S. Jin, O. Kwon: Effect of alloying elements on mechanical propeties and phase transformation of cold rolled TRIP steel steels, ISIJ International, 2001, vol. 41, No 3, pp. 290-297.

- [14]S. H. Park, W. Y Choo, N. J. Kim, J. H. Ko: Effects of hot rolling conditions on the microstructure and tensile properties of Nb-bearing TRIP steels, International Symposium on Hot Workability and Light Alloys Composites, TMS of CIM, Motrioll Quebek, 1996, Canada, pp. 493.
- [15] M. GRAF, S. Härtel, A. Bauer, W. Förster, D. Bublíková, M. Wagner, B. Awiszus, B. Mašek .: Development of a Quenching-Partitioning Process Chain for Forging Components, Materials Science Forum, 2018, Vol. 918, p. 89-92, https://doi.org/10.4028/www.scientific.net/MSF.918.85
- [16] V. Pileček, F. Vančura, H. Jirková, B. Mašek: Material-Technological Modelling of the Die Forging of 42CrMoS4 Steel, Materials and Technology, Vol. 48, 2014, p. 869-873
- [17] Vorel, F. Vančura, B. Mašek: Material-Technological Modelling of Controlled Cooling of Closed die Forgings from Finish Forging Temperature. In METAL 2015 24th International Conference on Metallurgy and Materials, Ostrava: 2015 TANGER Ltd., 2015, p. 202-208, ISBN: 978-80-87294-62-8
- [18] Š. Jeníček, I. Vorel, J. Káňa, K. Opatová: The Use of Material-Technological Modelling to Determine the Effect of Temperature and Amount of Deformation on Microstructure Evolution in a Closed-Die Forging Treated by Controlled Cooling, Manufacturing Technology, Vol. 17, 2017, p. 326-330
- [19] J. Káňa, I. Vorel, A. Ronešová: Simulator of Thermomechanical Treatment of Metals, In Daaam 2015. Vienna: Daaam International Vienna, 2016, p. 0513-05018, ISBN: 978-3-902734-07-5, ISSN: 1726-9679
- [20] B. Mašek, H. Jirková, J. Malina, L. Skálová, L. W. Meyer: Physical Modelling of Microstructure Development During Technological Processes with Intensive Incremental

Deformation, Key Engineering Materials, Vol. 345-346, 2007, p. 934-946, doi:10.4028 / www.scientific.net / KEM.345-346.943

# Acknowledgements

The present contribution has been prepared under project LO1502 'Development of the Regional Technological In-stitute' under the auspices of the National Sustainability Programme I of the Ministry of Education of the Czech Republic aimed to support research, experimental development and innovation.