DOI: 10.24425/amm.2021.134756

V. KAŇA[1*], V. KRUTIŠ[1], P. BOŘIL[1], A. ZÁDĚRA[1], M. RIMKO[1]

INFLUENCE OF HEAT TREATMENT AND NICKEL CONTENT ON THE PROPERTIES OF THE GX4CrNi13-4 STEEL

For the EN GX4CrNi13-4 martensitic stainless steel, research was conducted to investigate the impact of the quenching intensity and the content of nickel on the mechanical properties and amount of retained austenite. It was found that the amount of retained austenite significantly increases with growing nickel concentration. On the other hand, the cooling rate at quenching makes a difference only if the cooling is intensive, then amount of retained austenite decrease. A higher nickel content improves the mechanical properties. With more intensive cooling, the tensile strength decreases while the yield strength increases. The ductility is not significantly affected by the cooling intensity.

Keywords: Martensitic steel, Retained austenite, Mechanical properties, Heat treatment

1. Introduction

Martensitic stainless steel containing about 13% of chrome and low carbon was developed in Switzerland spreading rapidly all over the world. [1] It is standardized in all advanced countries of the world. In Europe, for example, it is regulated by the EN GX4CrNi13-4 or 1.4317 standard, in the United States by ASTM CA6NM, in Japan JIS SCS5, in UK by BS 425C11, and in France AFNOR Z6CN13-04. Basic structure consists of low-carbon martensite with a certain amount of retained austenite. This structure is achieved by heat treatment consisting of quenching and tempering. It is manufactured as both wrought and cast material. The present paper is concerned with the cast variety. This steel has excellent mechanical properties even at very low temperatures being well resistant to corrosion in different environments and its good weldability is also important. It finds its applications in hydropower engineering in turbine manufacturing, in oil and gas industry, in the manufacture of pumps, valves, and parts of pipeline systems [2,3].

2. Materials and methods

An experiment was conducted to assess the effects of the nickel content and the cooling rates in quenching on the mechanical properties of the EN GX4CrNi13-4 steel. To this end, two heats were carried out at the Brno University of Technology (BUT) university foundry each with a different nickel content. In the first heat, the content was chosen near the lower limit as given by the EN 10283 standard while, in the second one, it was near the upper limit. Either heat provided three test blocks 60 mm thick. The chemical composition of the heats is given in Table 1.

In each heat, two plates sized 60 × 110 × 21 mm were cut from the cast blocks to determine the effects of the heat treatment for each of the four selected quenching methods. Cutting plan of the test block is in Fig. 1. Each plate was marked by a four-digit number with the first three digits identifying the heat number, and thus the nickel content, and the last digit indicating how the

TABLE 1

Chemical composition of the heats

Heat no.	Element concentration [wt. %]										
	C	Si	Mn	Cr	Mo	Ni	Cu	N	Al	P	S
490	0.035	0.33	0.70	12.81	0.34	**3.59**	0.09	0.021	0.021	0.012	0.005
491	0.031	0.28	0.78	13.21	0.34	**5.00**	0.08	0.026	0.020	0.015	0.004

1 BRNO UNIVERSITY OF TECHNOLOGY, FACULTY OF MECHANICAL ENGINEERING, INSTITUTE OF MANUFACTURING TECHNOLOGY, DEPARTMENT OF FOUNDRY ENGINEERING, TECHNICKÁ 2896/2, BRNO 619 69, CZECH REPUBLIC

* Corresponding author: kana@fme.vutbr.cz

steel is cooled down from the initial temperature. The following modes were chosen:

- 1 – cooled in still air,
- 2 – cooled by air blown by a fan at about 4 m/s,
- 3 – cooled by air with water spray blown by a fan at about 4 m/s,
- 4 – cooled by immersing in water at the room temperature.

Example: marking 4912 denotes a sample from heat 491 (i.e., 5.00 % Ni) cooled by blowing air.

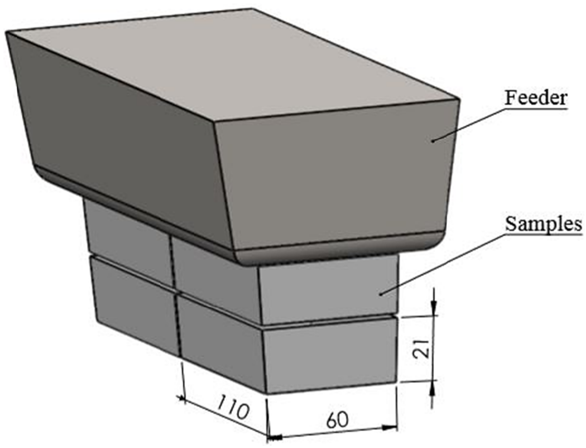

Fig. 1. Cutting plan of the test block

A hole with a diameter of 4 mm, about 45 mm deep was drilled in each testing plate to hold a type K thermocouple measuring the temperature in the plate to determine the cooling rate for each mode. The heat treatment consisted of heating to an austenitizing temperature of 1025°C holding it for one hour and, subsequently, quenching in one of the above modes. The quenching was then followed by tempering, the same for all the plates tested, namely 620°C for 2 h, cooled in furnace.

After the heat treatment, the plates were longitudinally cut into three identical parts. From either of the outer parts, a standard testing rod with a diameter of 10 mm was prepared for a tensile test. Thus, for each chemical composition and each heat treatment mode, two testing rods were tested, which makes a total of 16 testing rods. The tensile test was carried out using the Zwick/Roell Z250 universal machine equipped with the MultiXtens extensometer.

As the mechanical properties are closely related to the amount of retained austenite, it was measured by the XPert Panalytical diffractometer in the Bragg-Brentan arrangement, using Cu-Kα radiation.

3. Theory section

As mentioned above, the EN GX4CrNi13-4 steel has very good properties, particularly mechanical ones. The minimal properties required by EN 10283, i.e., *Rm* min. 760 MPa, $Rp_{0.2}$ min. 550 MPa, and A min. 15%, can only be achieved if the cast product has been heat- treated – quenched and tempered. The austenitizing temperature and delay at this temperature before quenching are given by the requirement to reach a homogeneous austenitic structure while preventing grain coarsening. The austenitizing temperatures found in the literature are mostly ranging between 1040°C and 1050°C [2-6], but some authors report both higher (1093°C [7]) and lower (950°C [1], 1100°C [8]) temperatures. For the present experiment, the temperature chosen was 1025°C. This steel is self-hardening, by [4], even when quenched in the air, the steel is fully hardened to a depth of up to 300 mm with its structure consisting of 100-percent martensite. Of course, the material will only achieve the required properties after tempering when retained austenite appears in the structure. It is then its quantity that directly determines its resulting properties. This quantity depends on the tempering temperature and holding at this temperature as well as on the chemical composition of the material. Generally, the highest possible amount of retained austenite is considered optimal, when the optimal combination is achieved of high ductility (about 20%) and good strength (R_m up to 900 MPa). The influence of the chemical composition on the material properties is only mentioned marginally in the literature [3,9]. It follows from the conclusions that a higher content of austenite-stabilizing elements, typically Ni, can lead to an increased amount of retained austenite. Papers abound in the literature on the tempering temperature and its influence on the mechanical properties of low-carbon martensitic steel with 13% of Cr. Many of them are concerned with the way the tempering temperature and holding time or repeated tempering affect the resulting material properties. The optimal tempering temperatures are mostly reported to range between 600°C and 650° C [1,2,4-7,9]. Thus, a tempering temperature of 620°C was chosen for this experiment. However, very little attention is paid by the literature to the influence of the quenching speed. This is the reason why this experiment was designed to analyze the impact of different quenching media on the mechanical properties and the resulting amount of retained austenite. Also the influence of two different values of the nickel content was analyzed, namely the minimal and maximal concentrations allowed by the EN 10283 standard for the EN GX4CrNi13-4 steel, which is 3.5 to 5.0% of Ni.

4. Results and discussion

A series of temperatures measured along the axis of the samples were recorded from thermocouples placed in the test plates. The cooling curves seen in Fig. 2 can be used to compare the cooling rates of the quenching media.

From such curves the average cooling rates between 400 and 100°C were found. This temperature range was chosen to contain the M_s and M_f temperatures of the tested steel type [4,5]. These cooling rates are shown in Table 2.

It can be seen that immersion in water resulted in an extremely rapid drop of temperature. From 400°C, the temperature dropped down to 100°C in 2 seconds.

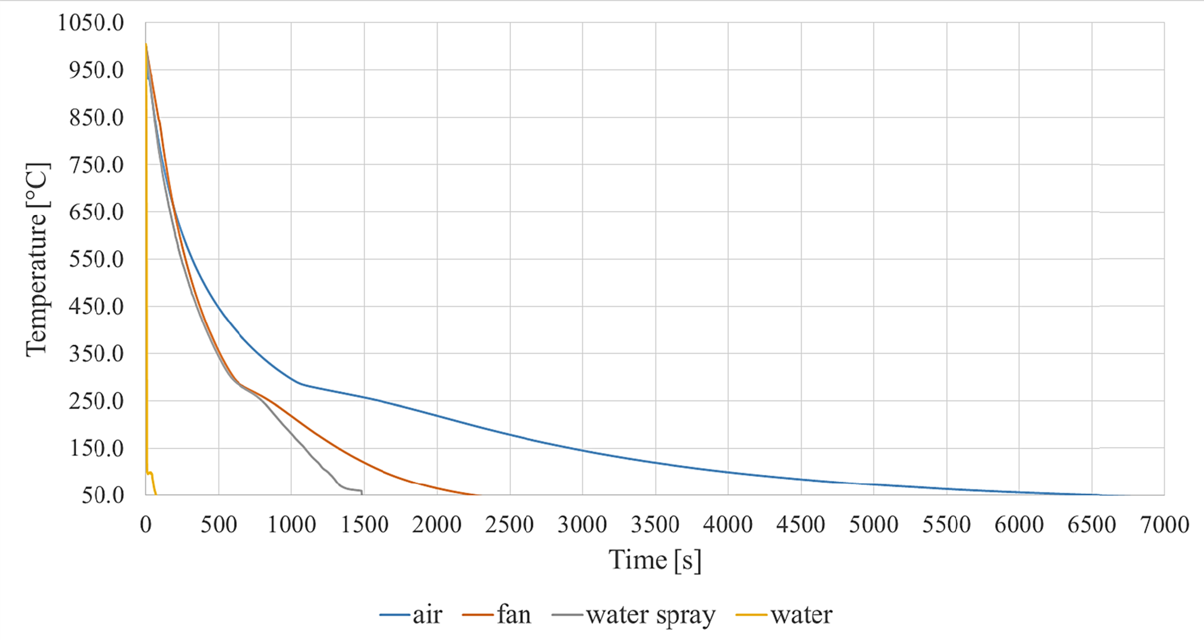

Fig. 2. Cooling curves along the axis of the sample

TABLE 2

Deducted cooling rates

Cooling medium	Air	Fan	Water spray	Water
Cooling rate [°C/s]	0.1	0.3	0.4	150.0
Cooling rate [°C/min]	5.4	15.0	21.6	9000.0

The completely heat treated samples were machined and a tensile test was carried out. Two test bars from each sample were tested. Its results are shown in Table 3.

TABLE 3

Results of the tensile test

Sample	$Rp_{0.2}$ [MPa]	*Rm* [MPa]	*A* [%]
EN 10283	min. 550	min. 760	min. 15
4901	609	786	19.5
	617	790	18.7
4902	628	782	19.3
	642	783	20.0
4903	673	784	15.9
	682	783	18.3
4904	682	758	19.8
	678	757	19.0
4911	667	814	20.0
	681	815	20.2
4912	618	804	19.8
	635	807	21.2
4913	693	808	19.9
	706	810	20.1
4914	686	783	21.2
	694	784	20.2

From the results of the tensile test it can be seen that, with an ample reserve, almost all samples meet the minimum mechanical property requirements of EN 10283. The exception is the tensile strength of sample 4904, but it is very tightly.

In the torn samples, the amount of retained austenite was then measured. The threaded ends of the test rods, not affected by plastic deformation, were used for this. The measured values are shown in Table 4.

TABLE 4

Measured values of the amount of retained austenite

Sample	4901	4902	4903	4904	4911	4912	4913	4914
Retained austenite [%]	14.5	14.4	13.9	5.9	36.8	37.3	36.1	24.6

The values indicate that the nickel content has a major influence on the amount of retained austenite. By the nickel content being increased from 3.59% to 5.00% the amount of retained austenite increased over 2.5 times. It can also be seen here that water cooling always resulted in a lower amount of the retained austenite. When other, less intensive, cooling methods were used, this amount never changed significantly.

The data shown in Table 3 and Table 4 are displayed more clearly structured in Fig. 3, Fig. 4 and Fig. 5. The values from Table 3 are averaged.

Fig. 3 suggest that, as the quenching cooling intensity grows, the yield strength of samples containing 3.59% of nickel tends to increase while, in samples containing 5.00% of nickel, this tendency was not confirmed. On the other hand, with the tensile strength in Fig. 4, increasing cooling rate resulted in a decreasing trend regardless of the nickel content. For ductility in Fig. 5, no significant changes were observed caused by changing cooling intensity. Samples containing 3.59% Ni showed an average elongation of 18.8% (standard deviation 1.0%) and

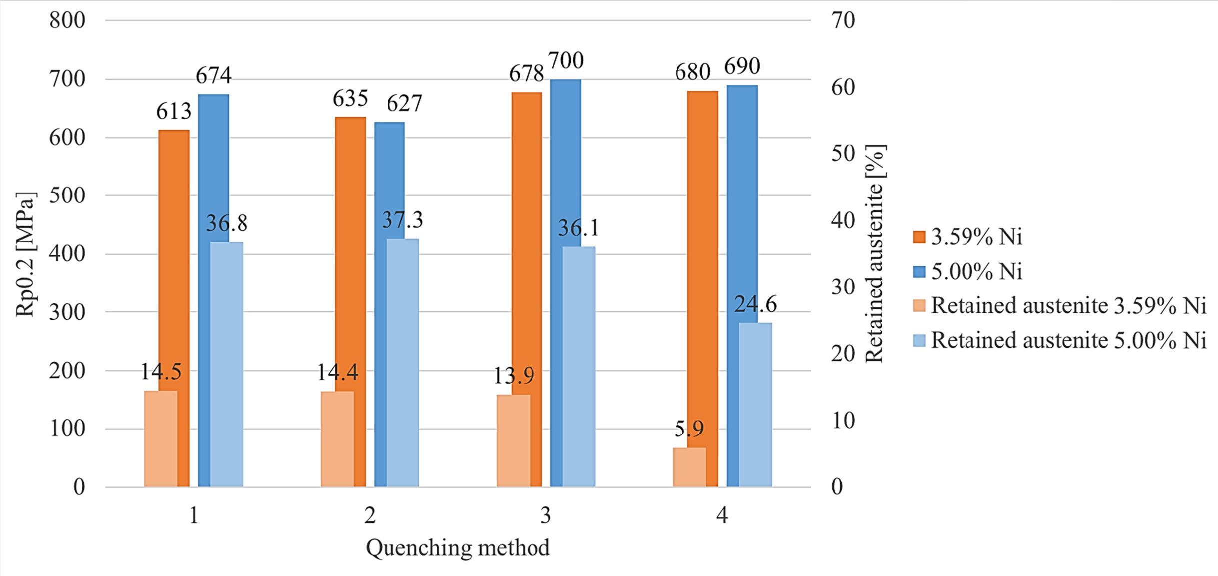

Fig. 3. Ni content and cooling intensity influencing $Rp_{0.2}$ and amount of retained austenite

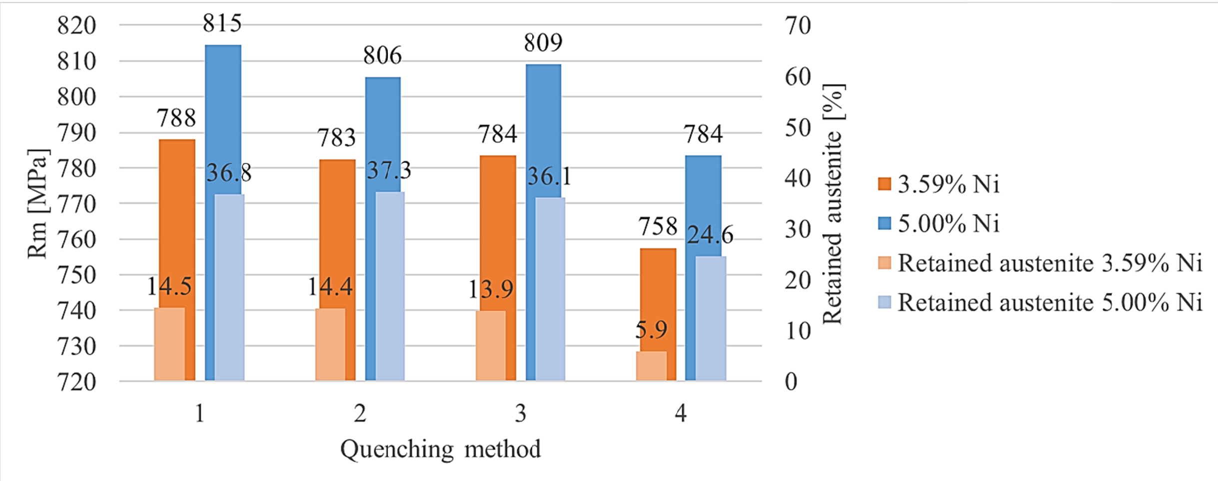

Fig. 4. Ni content and cooling intensity influencing *Rm* and amount of retained austenite

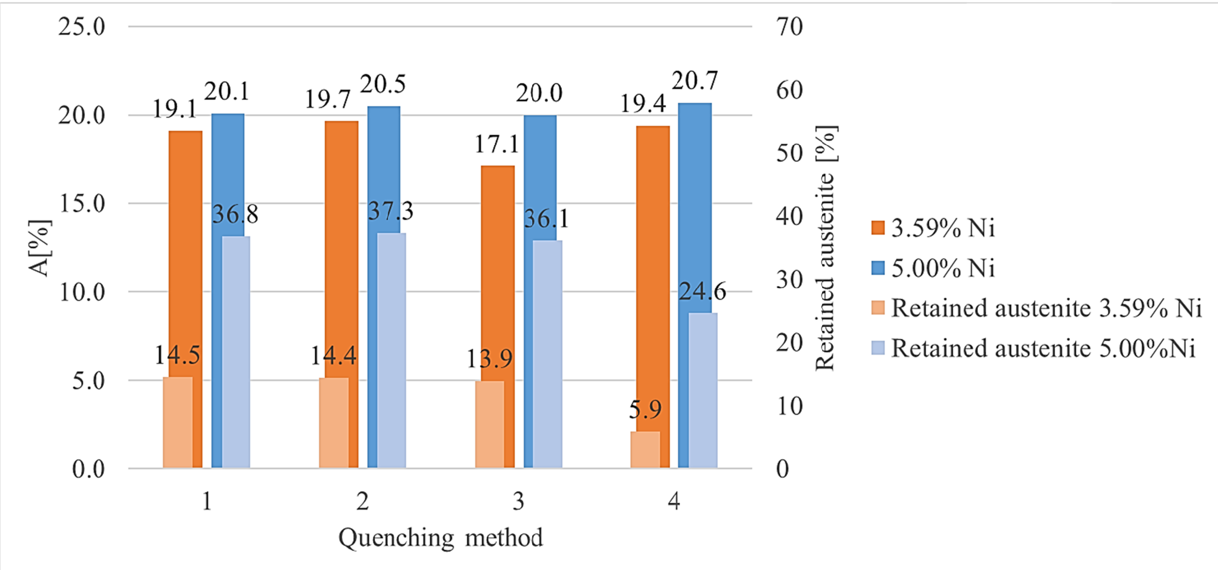

Fig. 5. Ni content and cooling intensity influencing ductility and amount of retained austenite

samples containing 5.00% Ni showed an average elongation of 20.3% (standard deviation 0.3%).

Concerning the influence of the nickel content on the mechanical properties, with the concentration varying within the range given by the standard, it does not seem to be the major importance. However, samples with a higher content of nickel, with a single exception, always had better mechanical properties. For example, the tensile strengths of all samples containing 5.00% of nickel, were about 25 MPa higher than in samples containing 3.59% of nickel. With the yield strength, this trend is not so unequivocal. In the event of the samples being cooled by air, using a fan with water spray and immersion in water, in materials with a higher nickel content, the measured yield strength values were by 10 to 61 MPa higher than those in the low-nickel content materials. However, in a sample cooled by air with fan without water spray, the opposite was true with the measured yield strength value being about 9 MPa lower. No reason being known for this property deterioration and it was not further investigated. Looking at the influence of nickel content on the ductility in Fig. 4, it can be seen that, here too, for all materials with a higher nickel content, the achieved values were higher. In materials where nickel concentration was near the upper limit given by the standard, the ductility was about 0.9 to 2.9% higher than in those where nickel concentration was at the lower limit.

5. Conclusion

An experiment was carried out to see how the cooling rates in quenching the EN GX4CrNi13-4 steel influence the mechanical properties and the amount of retained austenite after the heat treatment. The influence was investigated as well of the nickel content within the range given by the EN 10283 standard on the mechanical properties and the amount of retained austenite in this steel. The results of this experiment can be summarised as follows.

– The nickel concentration has a major impact on the amount of retained austenite. An increase of the Ni concentration from 3.59% to 5.00% causes amount of retained austenite to increase over 2.5 times.
– The influence of cooling rates during quenching is not much significant. Only in samples cooled by immersion in water, i.e. with the highest cooling intensity, significantly lower amounts of retained austenite were measured.
– The tensile strength slightly decreases with higher cooling rates regardless of the nickel content.
– On the other hand, in steel containing 3.59% of nickel, the yield strength slightly increases. No such trend, however, was observed in steel containing 5.00% of nickel.
– No influence was observed of the cooling rates on ductility.
– The nickel content varying within the range given by the EN 10283 standard (3.5 to 5.0%) has no influence on achieving the minimal required mechanical properties.
– Nevertheless, with nickel content near the upper limit given by the standard, the resulting mechanical properties are better. In tensile strength, for example, the difference was about 25 MPa.

Acknowledgement

This work was supported by the specific research project of BUT FME Brno, no. FSI-S-19-5981: Research of rapid prototyping using investment casting technology.

REFERENCES

[1] Y. Iwabuchy, JSME International Journal **46** (3), 441-446 (2003), DOI: 10.1299/jsmea.46.441.

[2] D. Zou, Y. Han, W. Zhang, X. Fang, Journal of Iron and Steel Research, International **17** (8), 50-54 (2010), DOI: 10.1016/S1006-706X(10)60128-8.

[3] Y.Y. Song, D.H. Ping, F.X. Yin, X.Y. Li, Y.Y. Li, Materials Science and Engineering A **527**, 614-618 (2010), DOI: 10.1016/j.msea.2009.08.022.

[4] https://www.researchgate.net/publication/280682597_Carbon_content_and_heat_treatment_effects_on_microstructures_and_mechanical_properties_of_13Cr-4Ni_martensitic_stainless_steel, accessed: 28.01.2020

[5] Y. Liu, D. Ye, Q. Yong, J. Su, K. Zhao, W. Jiang, Journal of Iron and Steel Research, International **18** (11), 60-66 (2011).

[6] Y. Song, X. Li, L. Rong, Y. Li, Materials Science and Engineering A **528**, 4075-4079 (2011).

[7] H. Niinaka, A. Hirose, S. Sogabe, H. Noguchi, Kawasaki steel technical report **No. 14**, 141-152 (1986).

[8] B.A. Tabatabae, F. Ashrafizadeh, A.M. Hassanli, ISIJ International **51** (3), 471-475 (2011).

[9] Y. Iwabuchi, Tetsu-to-Hagane (J. Iron Steel Inst. Jpn.) **70** (1), 120-127 (1984).