



# INFLUENCE OF AUSTENITIZING TEMPERATURE ON MICROSTRUCTURE AND KINETICS GROWTH OF AUSTENITE GRAIN IN 9 CRNB STEEL

PARILÁK Ľudovíť<sup>1,2</sup>, BEKEČ Pavel<sup>1\*</sup>, BERAXA Pavol<sup>1,2</sup>

<sup>1</sup>ŽP Research and Development Centre, Ltd., Podbrezová, Slovakia, EU, \*<u>bekec@zelpo.sk</u> <sup>2</sup>Faculty of Manufacturing Technologies with a Seat in Prešov, Slovakia, EU

## Abstract

This paper deals with the influence of various austenitizing temperatures on microstructure and kinetics growth of austenite grain in 9 CrNB steel. High austenitizing temperatures (1070 °C, 1150 °C, 1170 °C, 1190 °C, 1210 °C and 1230 °C) with a holding time 120 min were used. The microstructure was formed by martensite after austenitization. The occurrence of delta ferrite was observed in microstructure at temperature higher than 1190 °C. Delta ferrite was located at the grain boundaries and its amount was about 3 % at the austenitization temperatures of 1190 °C and 1210 °C and up to 5.5 % at the temperature of 1230 °C. In terms of the kinetics of austenite grain growth, it can be concluded, that up to temperature of 1170 °C there is an increase in grain size. At the higher austenitization temperature (over 1190 °C) we observed the decrease of grain size.

Keywords: Austenitizing temperature, heat treatment, delta ferrite, grain growth, low carbon 9Cr steel

## 1. INTRODUCTION

Low carbon 9CrNB steels contain 9% Cr alloying and have optimal ratio of B/N [1]. The choice of austenitization temperature is important from two aspects: temperature and alloy content. At the temperature perfect austenitization of solid solution must occur. This condition is fulfilled at the austenitization temperature, which is 30 - 50 °C above the temperature Ac<sub>3</sub>. In case of low carbon and low alloyed steels are the temperatures on the level at 920 - 940 °C. However in the case of high alloyed steel is needed to be also considered conditions of dissolution of the particles of secondary phases, which after the subsequent cooling forms the required secondary particles (precipitates). These significantly influence mechanical and creep properties of steel. From the chemical point of view, it is the formation of the carbide, carbo-nitride and nitride phases of the respective alloying elements (Cr, W, V, B) [2, 3]. Based on the knowledge of the dissolution diagrams and on the theoretical knowledge [4], the required austenitizing temperatures are above 1100 °C to about 1200 °C. Therefore were selected high austenitization temperatures of 1070 °C, 1150 °C, 1170°C, 1190 °C, 1210 °C and 1230 °C to achieve sufficient dissolution of carbide, carbo-nitride phases. The aim of this work was to focus on the kinetics growth of the austenite grains and microstructure at the selected austenitization temperature in steel.

# 2. MATERIAL AND EXPERIMENTAL METHODS

As the experimental material were used samples from 9 CrNB steel, that have been heat treated (austenitizing) at the temperatures of 1070 °C, 1150 °C, 1170 °C, 1190 °C, 1210 °C and 1230 °C with holding time 120 min. The chemical composition of 9CrNB is shown in **Table 1**. The samples were prepared by standard metallographic procedures (grinding, polishing, and etching). Microstructural analysis was carried out using an Olympus GX51 light optical microscope.



Elements	С	Mn	Si	Р	S	Cr	Ni	Мо	W	Со	В	Ν
Min [wt. %]	0.06	0.40	0.20	-	-	8.00	-	-	2.50	2.80	0.010	0.005
Max [wt. %]	0.10	0.50	0.35	0.020	0.008	9.00	0.15	0.10	3.00	3.20	0.015	0.015

#### Table 1 Chemical composition of 9CrNB steel

## 3. RESULTS

The microstructure in the conditions after austenitizing at selected temperatures was formed by martensite. This steel is air hardening. At the austenitization temperature of 1070 °C was microstructure homogenous, from point of view of austenitic grain size (**Figure 1**). The size of austenite grain ranged from 35 to 50  $\mu$ m and an average grain size was 43.1  $\mu$ m. At the temperature of 1150 °C was seldom observed heterogeneity of grain size (**Figure 2**). The size ranged of approximately from 90 to 190  $\mu$ m, the average size reached 138.6  $\mu$ m.





Figure 1 Microstructure at the austenitization temperature 1070 °C (martenzite)

Figure 2 Microstructure at the austenitization temperature 1150 °C (martenzite)



Figure 3 Microstructure at the austenitization temperature 1170 °C (martenzite)



**Figure 4** Microstructure at the austenitization temperature 1190 °C (martenzite + delta ferrite)

The microstructure at temperature of 1170 °C was coarser-grained in comparison with the lower temperatures. It was also observed slight heterogeneity of austenite grain sizes (**Figure 3**). The grain sizes were in the range of about 110 - 230  $\mu$ m. The average size of grain was 172.3  $\mu$ m. At the temperature of 1170 °C in the



microstructure was an also small amount of delta ferrite observed. Delta ferrite was excluded at the austenite grain boundaries. The amount of delta ferrite in the microstructure was negligible. At the temperature of 1190 °C larger amount of delta ferrite is observed (approximately 3%) in comparison to temperature of 1170 °C, which was excluded at the austenite grain boundaries (**Figure 4**). Microstructure was slightly heterogeneous; the grain size range was about 100 - 210  $\mu$ m and an average grain size reached 139.8  $\mu$ m. The formations of delta ferrite are larger, in comparison to the formations at the temperature 1170 °C. In comparison to the grain size at temperature of 1170 °C, grain size of the austenite decreased.

At the austenitization temperature of 1210 °C was similarly as at temperature 1190 °C in microstructure observed delta ferrite at the austenite grain boundaries (**Figure 5**). The amount of delta ferrite was comparable to that at temperature 1190 °C. Microstructure was relatively homogeneous, the grain size was at range of 100 - 160  $\mu$ m and the average grain size reached 131.2  $\mu$ m. At the highest temperature (1230 °C) was in the microstructure the highest occurrence of delta ferrite (5.5 %) observed, that was excluded at the austenite grain boundaries (**Figure 6**). The formations of delta ferrite were at this austenitization temperatures greatest. At the holding time 120 min was microstructure relatively homogeneous. The grain size was in the range from 80 to 120  $\mu$ m. The average austenitic grain size was 99.2  $\mu$ m.



**Figure 5** Microstructure at the austenitization temperature 1210 °C (martenzite + delta ferrite)

**Figure 6** Microstructure at the austenitization temperature 1230 °C (martenzite + delta ferrite)



Influence of austenitizing temperature on growth grain - holding time 120 min.

Figure 7 The influence of austenitizing temperature on grain growth at the holding time of 120 min



**Figure 7** shows influence of austenitizing temperature on the grain growth at the holding time 120 min. As can be seen, under the temperature of 1170 °C, the grain size gradually increases. Above temperature of 1190 °C, with the growth of temperature are austenitic grains smaller with the same increase of volume fraction of delta ferrite in the microstructure.

## 4. DISCUSSION

The main objective of this work, as already mentioned in the introduction, was to focus on the growth kinetics of austenite grain and state of microstructure at the selected austenitizing temperature in the 9CrNB steel. The microstructure was formed by lath martensite. At the temperatures of 1070 °C and 1150 °C was microstructure quite homogeneous. At the higher temperatures of 1170 °C, 1190 °C and 1210 °C were observed slight heterogeneity in the microstructure. From the view of the kinetics of austenitic grain growth can be concluded, that increasing austenitizing temperature leads to increase of austenite grains size. But this applies only until the austenitizing temperature of 1170 °C. At higher temperatures of 1190 °C and 1210 °C the grain growth already did not occur, even occurred decreased of grain size. The reason was probably the presence of delta ferrite in the microstructure, which was excluded mainly on the grain boundaries. Due to this excluded delta ferrite, the grain growth was reduced and possibly to refinement of austenite grain size.

At the highest austenitizing temperature of 1230 °C, where was observed highest occurrence of delta ferrite (5.5 %), there was observed the second finest-particle microstructure. Delta ferrite is according to the binary equilibrium diagram of Fe - Fe<sub>3</sub>C stable at high temperatures (above 1396 °C). At the certain ratio of austenite forming and ferrite forming elements are this high temperature phase becomes stable at ambient temperatures. Delta ferrite may be desirable in some steels (duplex steels), but in the most cases it is undesirable. At the small content may negatively affect the mechanical properties of steel. 9 CrNB steel is the creep resistant steel alloyed mainly with Cr, W, Co, and with optimal ratio of B / N [5, 6]. In addition to these elements are in steel presented small amounts of Mn, C, Si, Ni, V. In the **Figure 8** calculated phase equilibrium composition of the steel MarBN in relation to the temperature are seen. The figure shows that the delta ferrite starts to equilibrate in the temperature of 1280 °C [7]. However in microscale and at the grain boundaries, where can be local enrichment (segregation) of ferrite forming elements, may result in the formation of delta ferrite at lower temperatures and to remain stable in non-equilibrium in the microstructure after cooling.



Figure 8 Thermodynamic equilibrium calculations of MarBN steel using the software MatCalc [7]



#### 5. CONCLUSION

From the obtained results of 9CrNB steel can be stated the following conclusions:

- The microstructure after austenitization was formed by martensite. At the temperature of 1170 °C was in the microstructure observed negligible occurrence of delta ferrite. At higher austenitization temperatures (1190 °C, 1210 °C) the proportion of delta ferrite in the microstructure increases (about 3 %). At the highest austenitization temperature of 1230 °C the proportion of delta ferrite was in microstructure about 5.5 %.
- 2) With increasing austenitizing temperature, up to the temperature of 1170 °C, the austenite grain size increases. At the higher austenitization temperatures from 1190 °C the increase of the grain size practically does not occur (grain size even decreases). It is probably due to the presence of delta ferrite in the microstructure, which was located at the grain boundaries.
- 3) From the point of view of growth kinetics of austenitic grain it can be concluded that 9 CrNB steel has local maximum in the range temperature about 1170 1180 °C. The question then remains, whether the austenitic grain refinement at higher austenitizing temperatures causes the presence of delta ferrite at the grain boundaries, or the exclusion of carbide phases that prevent grain growth. This will be the subject of further research.

#### ACKNOWLEDGEMENTS

# This work was supported by the project APVV-15-0723 (CREEPWELD) - Welding technology development for unique creep resistant steels currenly developed in Železiarne Podbrezová, a.s.

#### REFERENCES

- [1] SAWADA, K., TANEIKE, M., KIMURA, K., ABE, F. Effect of Nitrogen Content on Microstructural Aspects and Creep Behavior in Extremely Low Carbon 9 Cr Heat-resistant Steel. *ISIJ International*, 2004, vol. 44, pp.1243-1249.
- [2] BEKEČ, P., PARILÁK, Ľ., BERAXA, P., MOJŽIŠ, M., DOMOVCOVÁ, L., FUJDA, M. Microstructure and Mechanical Properties of 9CrNB Steel after Heat Treatment. *Materials Science Forum*, 2017, vol. 891, pp.167-170.
- [3] OKADA, H., MUNEKI, S., YAMADA, K., OKUBO, H., IGARASHI, M., ABE, F. Effects of Alloying Elements on Creep Properties of 9Cr-3.3W-0.5Pd-V, Nb, N, B Steel. *ISIJ International*, 2002, vol. 42, pp.1169-1174.
- [4] BARNARD, P., MOODY, P., MACLACHLAN, Y., ALLEN, D., ROBERT, S., DU, H., THOMSON, R. A New MarBN Alloy for USC Power Plant. In Proc. of the 5<sup>th</sup> Symposium on Heat Resistant Steels and Alloys for High Efficiency USC/A-USC Power Plants 2013. Seoul: 2013, p.31.
- [5] DUDOVA, N., MISHNEV, R., KAIBYSHEV, R. Effect of Tempering on Microstructure and Mechanical Properties of Boron Containing 10% Cr Steel. *ISIJ International*, 2011, vol. 51, pp.1912-1918.
- [6] BAEK, J. W., NAM, S. W., KONG, B. O., RYU, S. H.: The Effect of Delta-Ferrite in P92 Steel On The Formation of Laves Phase and Cavities for The Reduction of Low Cycle Fatigue and Creep Fatigue Life. *Key Engineering Materials*, 2005, pp. 463-470.
- [7] SOMMITSCH, Ch., VANSTONE, R., KERN, T. U., BARNARD, P., MAYR, P., THOMSON, R., AGÜERO, A. Co-Ordination of Europe Research in Structural Materials for Power Generation Equipment. In *10th Liege Conference on Materials for Advanced Power Engineering 2014*, Liege: J. Lecomte-Beckers, O. Dedry, J. Oakey and B. Kuhn, 2014, pp.3-18.