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THE KINETICS OF AUSTENITE GRAIN GROWTH IN STEEL FOR WIND POWER PLANT SHAFTS

KINETYKA ROZROSTU ZIARNA AUSTENITU W STALI NA WAŁY SIŁOWNI WIATROWYCH

The results of investigations of the influence of the temperature and time of austenitising on the austenite grain size in 34CrNiMo6 steel – are presented in the hereby paper. This type of steel is used for wind power plant shafts. Determination of the austenite grain growth kinetics enables designing the heat treatment technology of large forgings in such a way as to obtain uniform and small grains. The temperature and time ranges, which can lead to the abnormal austenite grain growth and in consequence to qualitative worsening of forgings (introduction of the so-called structural notches) were determined. When discussing the reasons of the abnormal grain growth the simulation of the carbonitrides dissolving process was taken into account.

Keywords: austenite, grain size, wind power plant shaft, lowalloy steel, carbonitrides

W pracy przedstawiono wyniki badań wpływu temperatury i czasu austenityzowania na wielkość ziarna austenitu w stali 34CrNiMo6. Jest to stal stosowana na wały siłowni wiatrowych. Określenie kinetyki wzrostu ziarna austenitu pozwala na projektowanie technologii obróbki cieplnej dużych odkuwek tak aby uzyskać możliwie drobne, równomierne ziarno. Określono zakresy temperatur i czasów austenityzowania, które mogą prowadzić do anormalnego rozrostu ziarna austenitu a w konsekwencji do pogorszenia własności odkuwki (wprowadzenia tzw. karbów strukturalnych). W dyskusji przyczyn wystąpienia anormalnego rozrostu ziarna uwzględniono symulację procesu rozpuszczania węglikoazotków.

1. Introduction

Controlling of the influence of a temperature and time of austenitizing on the size and morphology of austenite grains is essential in industrial practice, because of the properties of forgings (e.g. crack resistance). According to Atkinson [1] two main features characterising a normal grain growth are: homogeneity of size and shape contained within a narrow interval and a time invariable size distribution – distribution maintains a similar shape, while the modal value increases with time. Temperature and time as well as contents and dispersion degree of precipitates inhibiting movement of grain boundaries at the increased temperatures are the basic parameters influencing austenite grain sizes. When precipitates inhibiting grain growth are not present, the influence of time τ and temperature T on an average diameter of austenite grain D is determined by Equation [2]:

$$D^2 - D_0^2 = A \exp\left[-\frac{Q}{RT}\right] \cdot \tau, \qquad (1)$$

where: D_0 – initial diameter of the austenite grain, Q – activation energy of grain growth, R – gas constant and A – constant. It is assumed, that the activation energy of grain growth is equal the activation energy of iron self-diffusion.

In the presence of precipitates inhibiting grain growth an average radius of austenite grains R_a depends on an average size of precipitates r as well as on a volume fraction of precipitates V_V, according to the Smith-Zener equation [3]:

$$R_a = \frac{4}{3}\frac{r}{V_V}, \qquad (2)$$

A problem of the austenite grain growth is complicated (especially in reference to alloy steels) and still discussed in the literature [4-18]. The chemical composition of steel influences the austenite grain growth in both cases, i.e. when there are no precipitates giving the grain boundary pinning effect – Equation (1) and when those precipitates are present (carbides, nitrides, carbonitrides) - Equation (2). In the first case this influ-

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ence is related to the interaction of elements, especially intersticial elements (C, N) dissolved in austenite, with the activation energy of iron self-diffusion [2,19]. In the second case the chemical composition of steel influences the temperature of dissolving the grain growth inhibitor and its tendency for coagulation [20,21].

The determination of changes in the grain size in dependence of a temperature and time of austenitizing of 34CrNiMo6 steel used for constructing shafts for wind power plants – is the aim of the presented paper.

2. Material for testing

34CrNiMo6 steel (acc. to PN-EN 10083) used for production of shafts for wind power plants was the tested material. The chemical composition of the tested steel is shown in Table 1. Mi-crostructure of this steel, as supplied, is presented in Figure 1.

TABLE 1

Chemical composition of the investigated steel

C	Mn	Si	P	S	Cr	Ni	Mo	V	Al	N	Fe
0.4	0.67	0.2	0.009	0.007	1.54	1.45	0.27	0.057	0.0098	0.005	Bal.

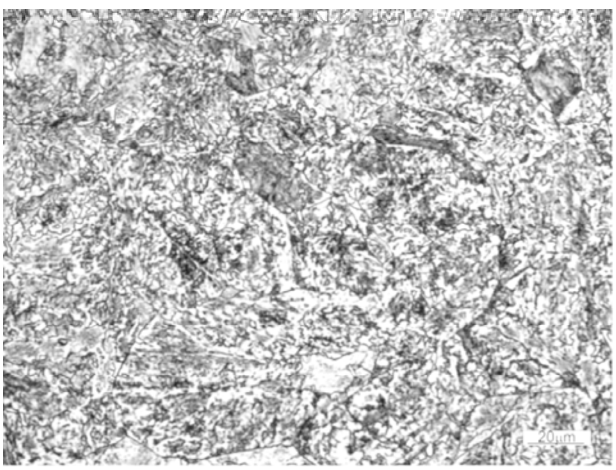

Fig. 1. Microstructure of investigated steels (as-delivered conditions). Etched with 2% nital

3. Experimental procedure

Temperatures: Ac_{1s} equal 740 °C and Ac_3 equal 780 °C were determined for the investigated steel by means of dilatometric tests. Basing on these characteristic temperatures, and applying the principle, that the selected austenitizing temperature for hardening must be 50 °C above Ac_3, it was decided that the optimal austenitizing temperature for 34CrNiMo6 steel should be 830 °C.

However, a temperature of 830 °C can be too low for the technological conditions (size of forging), especially due to changes in the chemical composition of the tested steel – being within permissible differences determined by the standard PN-EN 10083 – can influence changing of this temperature in a small degree. Therefore 840 °C was assumed as the lowest austenitizing temperature for the grain size determination. The next austenitizing temperature was determined on the basis of the technology applied in industrial conditions, and it was 870 °C. As the subsequent austenitizing temperatures of 900, 950, 1000 and 1100 °C were assumed.

The shortest austenitizing time was selected on the basis of the heat treatment methodology performed under the laboratory conditions on samples of a cubic shape with a side of 25 mm. This time was 20 minutes. Then 2, 7 and 24 hours were assumed as the remaining times. After the austenitizing samples were quenched in oil.

Metalographic microsections were prepared after the heat treatment. In order to attain microobsections the samples were cut at the half of thickness and the obtained surface ground on abrasive papers of grades from 150 to 2000. The successive step constituted polishing the surface with the use of water suspension Al_2O_3 and chemical etching to reveal grain boundaries of the prior austenite. Etching was done by means of a saturated water solution of picric acid with addition of a softening agent [20]. Optical microscope Axiovert 200 MAT of the Carl Zeiss Company was used for the observation of etched samples surfaces. Images of microstructure were recorded in order to perform the quantitative analysis. The austenite grain size was estimated on the basis of measurements of the length of chords of the austenite grains cut by random secants superimposed on the microstructure image. The length of chords was measured by means of the computer software SigmaScan Pro.

The contents of undissolved precipitates of carbonitrides V(C, N) as a function of the austenitizing temperature was calculated by means of the computer software Carbnit [21].

4. Results and discussion

Examples of microstructures of the samples after the heat treatment with revealed grain boundaries of the prior austenite are presented in Figures 2-7.

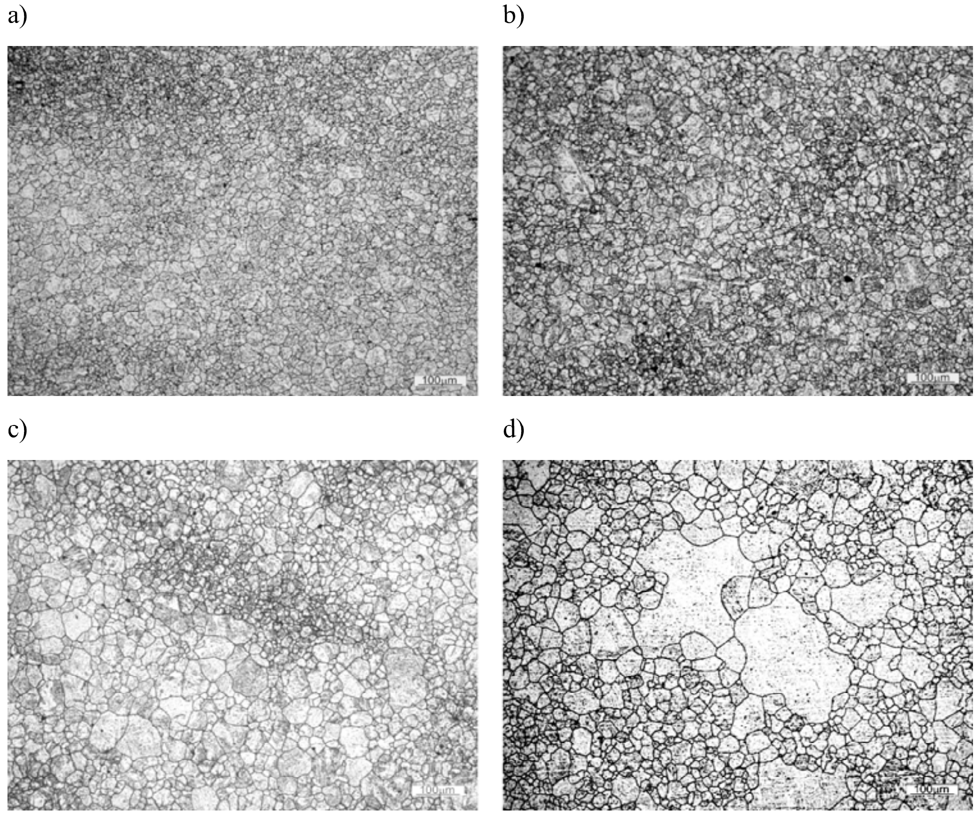

Fig. 2. Microstructures with revealed the prior austenite grain boundaries of samples quenched from a temperature of 840 °C and austenitised for: a) 20 min., b) 2 h, c) 7 h, d) 24 h

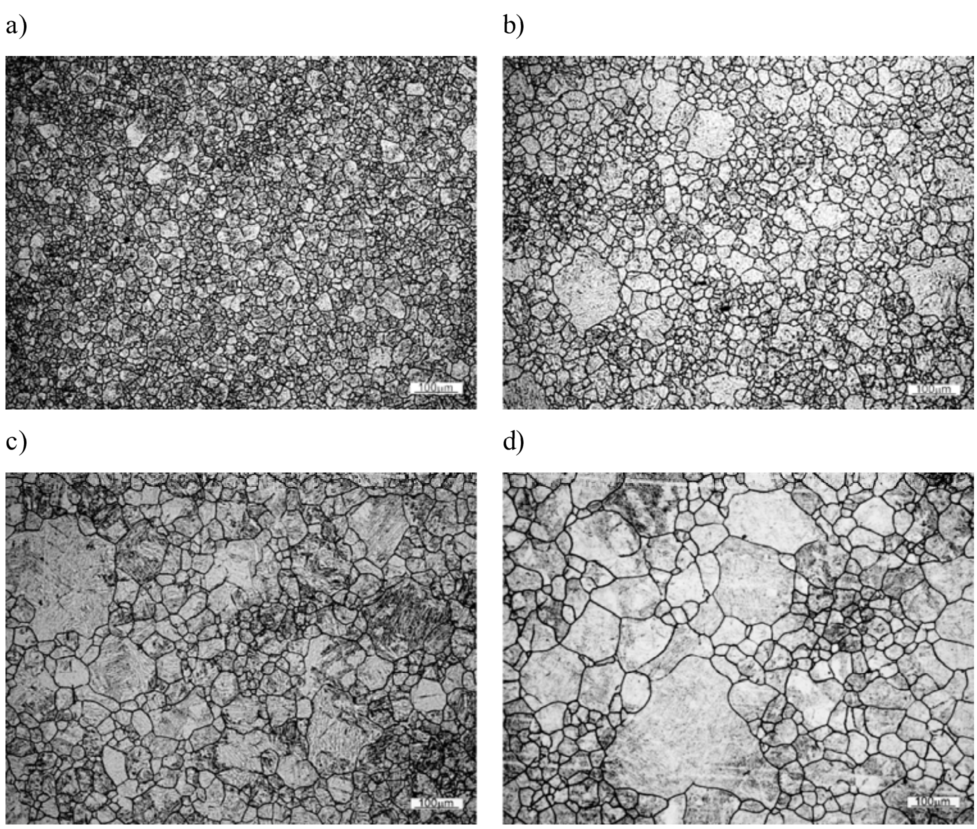

Fig. 3. Microstructures with revealed the prior austenite grain boundaries of samples quenched from a temperature of 870 °C and austenitised for: a) 20 min., b) 2 h, c) 7 h, d) 24 h

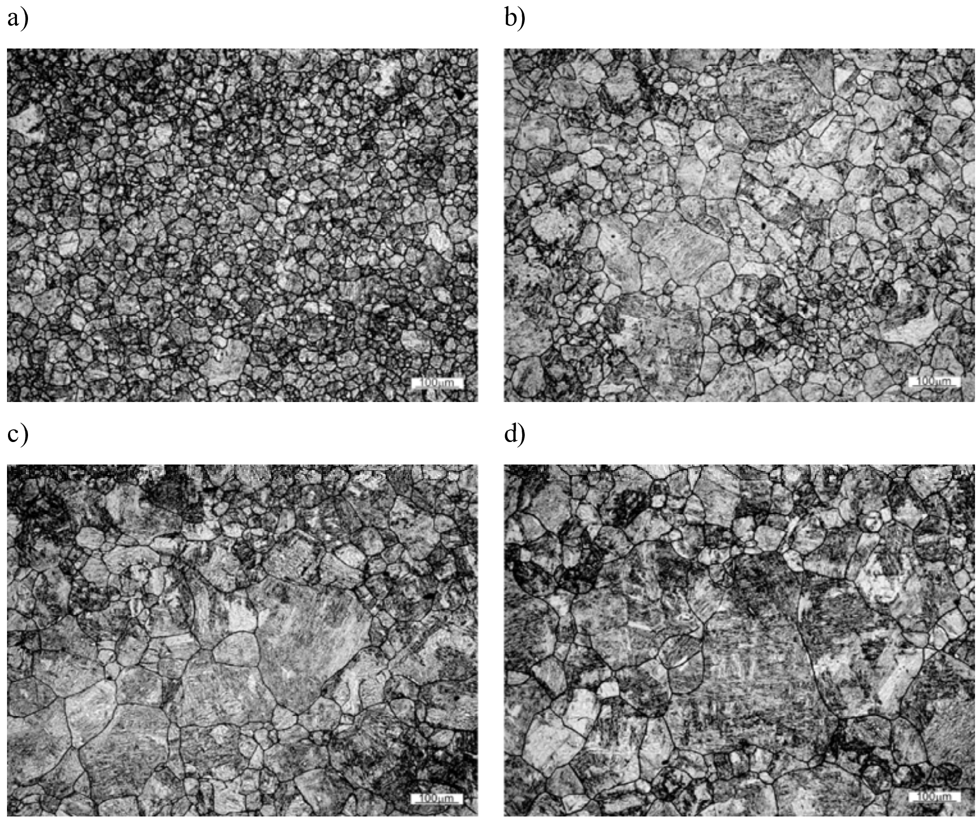

Fig. 4. Microstructures with revealed the prior austenite grain boundaries of samples quenched from a temperature of 900 °C and austenitised for: a) 20 min., b) 2 h, c) 7 h, d) 24 h

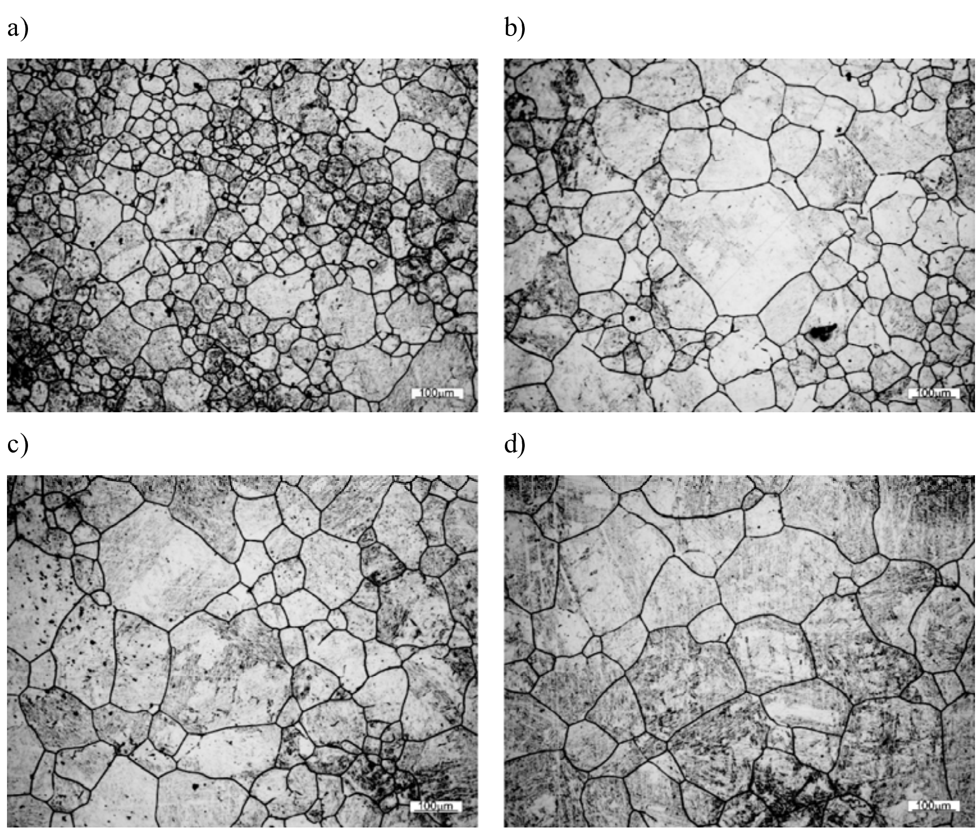

Fig. 5. Microstructures with revealed the prior austenite grain boundaries of samples quenched from a temperature of 950 °C and austenitised for: a) 20 min., b) 2 h, c) 7 h, d) 24 h

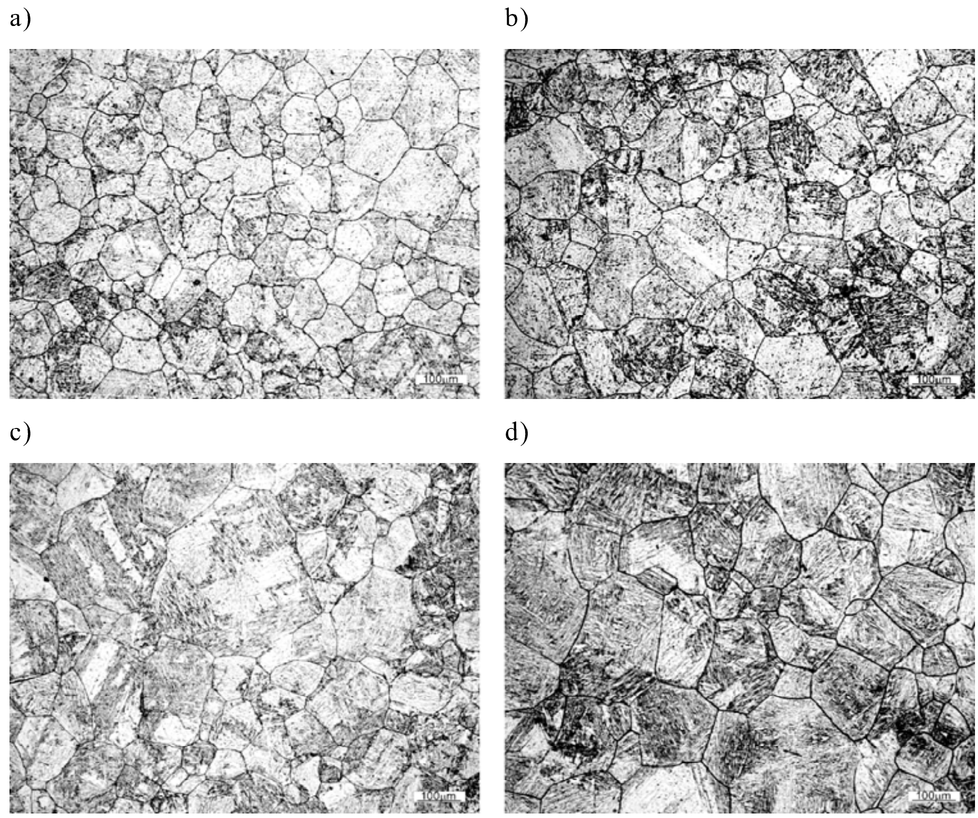

Fig. 6. Microstructures with revealed the prior austenite grain boundaries of samples quenched from a temperature of 1000 °C and austenitised for: a) 20 min., b) 2 h, c) 7 h, d) 24 h

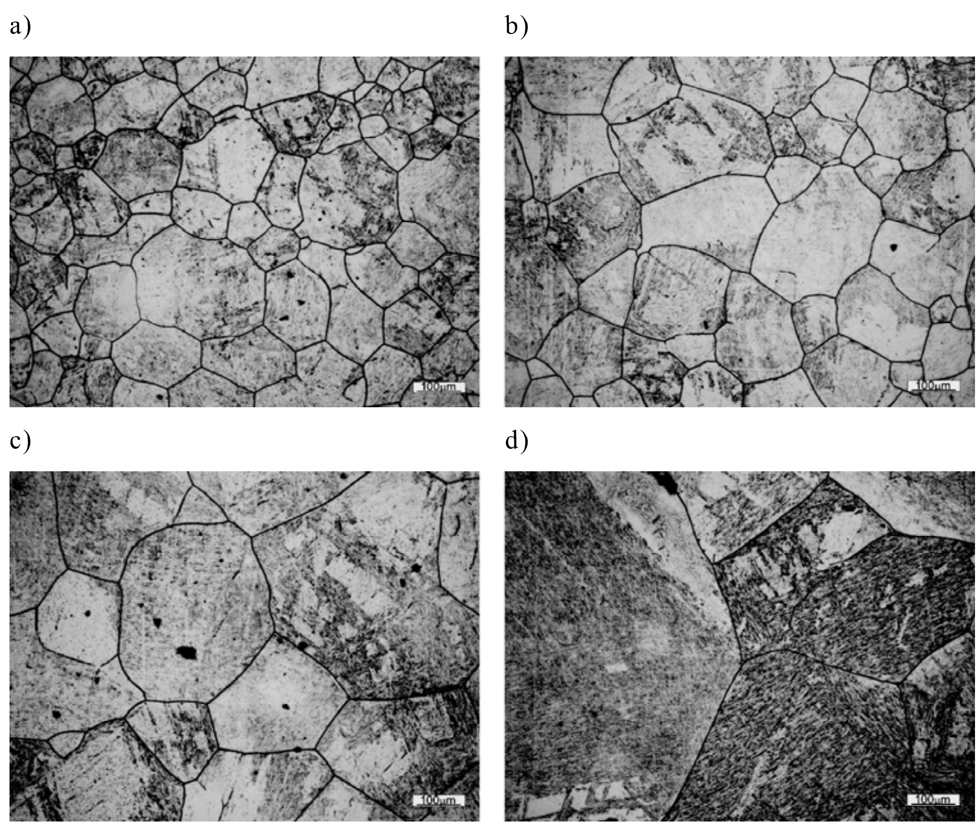

Fig. 7. Microstructures with revealed the prior austenite grain boundaries of samples quenched from a temperature of 1100 °C and austenitised for: a) 20 min., b) 2 h, c) 7 h, d) 24 h

The results of the quantitative analysis of the austenite grain size are given in Table 2. The dependence of the average chord length *l* and the austenite grain size on the austenitizing temperature and time is presented in Figure 8.

TABLE 2

The measurement results of the average grain chord length and the calculated grain size grade, acc. ASTM

Quenching temperature, °C	Austenitizing time, h	Average chord length *l* [μm]	Standard deviation [μm]	Calculated grain size grade G, acc. ASTM	Variability parameter
840	0.33	17	9	8.3	0.54
	2	24	13	7.4	0.54
	7	33	22	6.4	0.65
	24	36	32	6.2	0.88
870	0.33	25	12	7.3	0.48
	2	30	16	6.8	0.53
	7	46	33	5.5	0.72
	24	55	48	5.0	0.87
900	0.33	30	14	6.7	0.46
	2	45	30	5.6	0.67
	7	66	45	4.5	0.68
	24	88	53	3.6	0.60
950	0.33	54	30	5.1	0.56
	2	83	42	3.8	0.50
	7	96	48	3.4	0.50
	24	119	72	2.8	0.61
1000	0.33	78	42	4.0	0.54
	2	105	55	3.1	0.52
	7	117	63	2.8	0.54
	24	141	74	2.3	0.53
1100	0.33	119	60	2.8	0.51
	2	174	90	1.7	0.52
	7	265	137	0.5	0.52
	24	387	212	-0.6	0.55

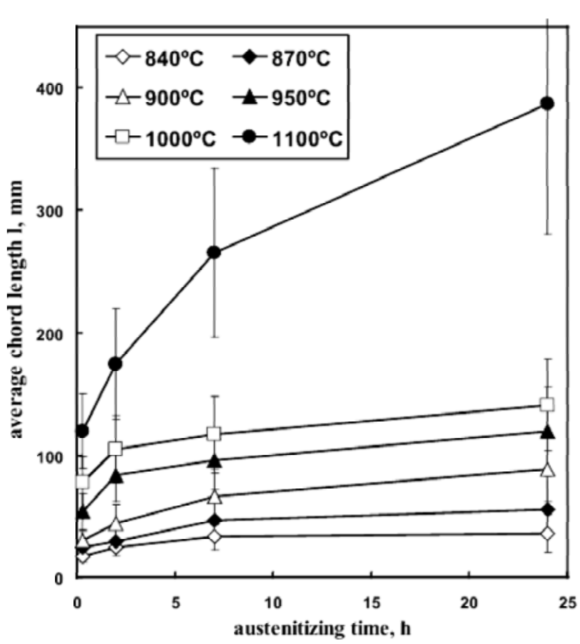

Fig. 8. Average chord length l versus the austenitization time

It can be seen, that with the increasing temperature the austenitizing time more strongly influences the grain size change. The lower temperature range (applied in an industrial practice) the stronger temperature influence.

The tested steel austenitized at 840 °C for 7 hours and at 900 °C for 20 minutes is characterised by practically the same grain size. Similar situation occurs after austenitizing at 870 °C for 24 hours and at 950 °C for 20 minutes. This is obvious, since – as can be seen in Equation (1) – the influence of a temperature on a grain size is exponential, while the influence of time is proportional to the square root of this parameter. However, it should be mentioned, that regardless of the similar grain size *G*, for the considered variants of heat treatments, there is an essential difference in microstructures between samples austenitized longer at a lower temperature and the ones austenitized for only 20 minutes but at temperatures of 900 and 950 °C. In both cases of the prolonged austenitizing time (840 °C/24 hs and 870 °C/24 hs) the abnormal growth of individual grains can be clearly observed (Fig. 2d and 3d). Data from Table 2 allowed to determine the so-called variability parameter (the ratio of the standard deviation to the average chord length *l*). It was assumed that normal grain growth occurs when this parameter value is larger or equal 0.6. The microstructure characterised with such variability parameter the most probably will be disadvantageous for the steel impact strength after a heat treatment.

Apart from effects related to a nucleation intensity, grain growth rate and heterogeneity of microstructure and the chemical composition, an abnormal grain growth – at prolonged austenitizing times in lower temperatures – can be related to coagulation processes of carbonitride V(C,N) precipitates blocking the austenite grain growth. The dependence of the calculated content of carbonitride V_V precipitates on temperature T is presented in Figure 9. A volume fraction of dissolved precipitates at a temperature of 800 °C equals 0.17 % and lowers with the increase of the austenitizing temperature. At a temperature of 1000°C, V_V=0.04 %. The calculated dissolution temperature of V(C,N) equals 1230 °C. At high austenitizing temperatures a function of precipitates in inhibiting the grain growth disappears since their percentage is nearing 0. At lower austenitizing temperatures the inhibiting effect of precipitates on the grain growth decreases with an increase of austenitizing time due to the coagulation of precipitates V(C,N). That time the abnormal grain growth can happen. At high temperatures (lower than the temperature of carbonitride dissolution) at a prolonged austenitizing time precipitates also coagulate, however their density (number in a volumetric unit) is so small, that the grain growth is in an accordance with Equation (1).

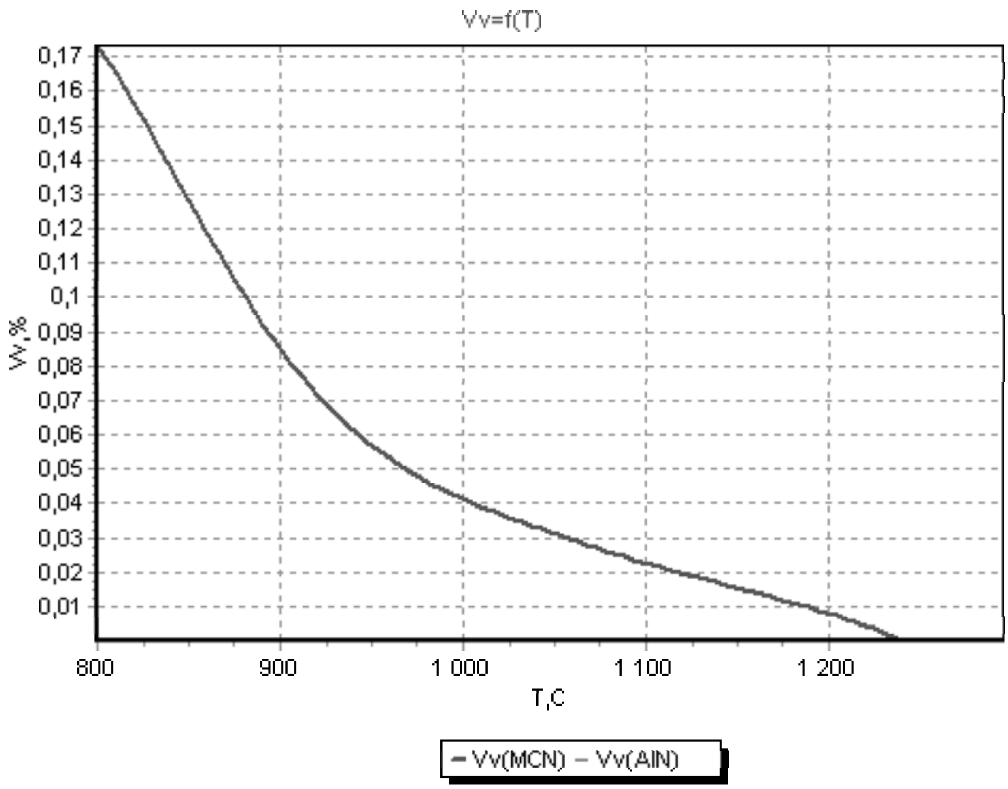

Fig. 9. Volume fraction of carbonitride precipitations (V_V) as a temperature function

Due to the required steel properties it is essential not to perform its austenitizing in such time and at such temperature when the so-called abnormal grain growth occurs.

The schematic presentation of the austenite grain growth kinetics in dependence of the austenitizing temperature was constructed (Fig. 10). The grain size is changing from No 9 (at a tem-perature of 840 °C and austenitizing time being 20 min.) to –1 (at a temperature of 1100 °C and austenitizing time of 24 hours). Applying the above given criterion of the determination of the abnormal grain growth the zone where it occurs is marked on the scheme in Figure 10b. Measurements of austenite grain sizes allowed to state, that the zones of parameters *T* and τ – at which the abnormal austenite grain growth occurs – seem to confirm the assumption concerning the role played by the coagulating precipitates V(C,N) in this process.

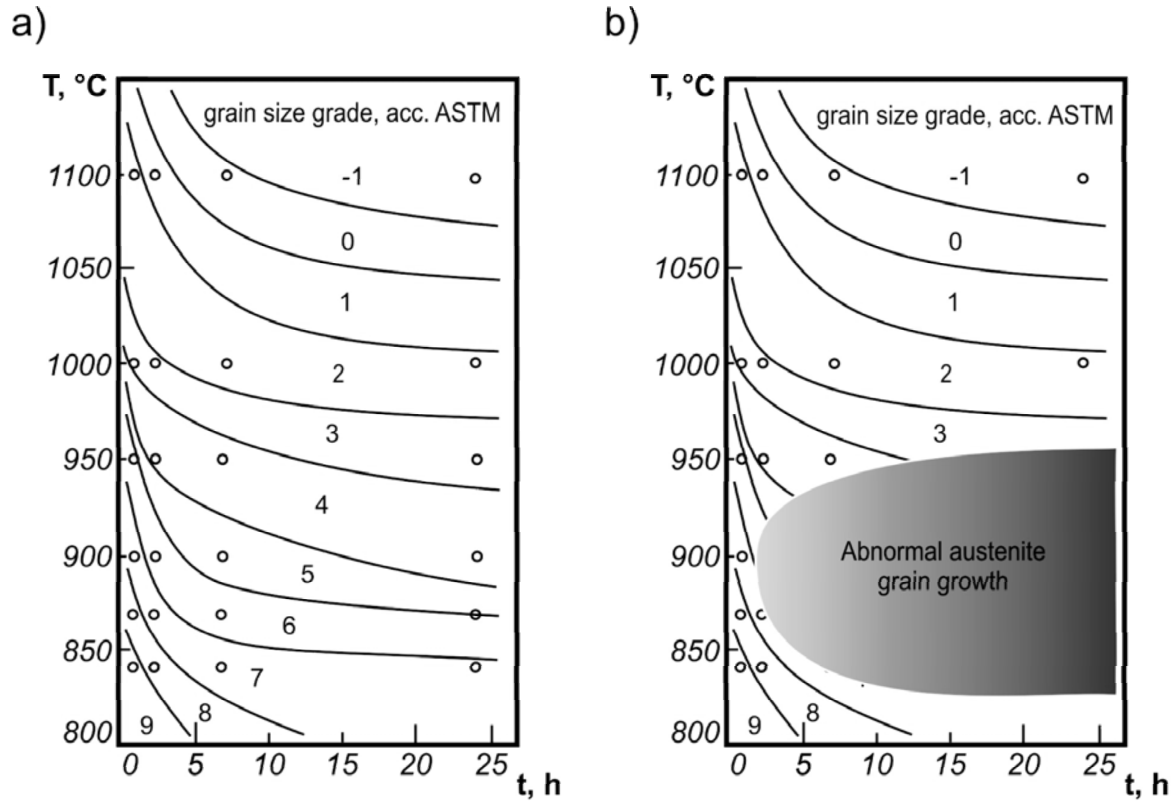

Fig. 10. Scheme constructed on the bases of data from Table 2: a) Kinetics of the austenite grain growth in 34CrNiMo6 steel as a temperature and time function, b) Fig 10a with the marked zone of the abnormal grain growth

5. Conclusions

Investigations carried out in the presented here work enabled to determine the influence of time and temperature of austenization on the size and morphology of the austenite grains. The obtained results allow to formulate the following conclusions:

- Increase of the austenitization temperature influences more strongly the austenite grain growth than the prolongation of time of this process.
- Prolongation of the austenitizing time (for the strictly determined austenitizing temperature range, not higher than 950 °C) can cause the abnormal grain growth.
- Abnormal austenite grain growth is probably related to the coagulation of carbonitride precipitates V(C,N).

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REFERENCES

[1] H. V. Atkinson, Theories of normal grain growth in pure single phase systems, Acta Metall. **36**, 469-490 (1988).
[2] W. C. Leslie, The physical metallurgy of steels, Mc Graw Hill Inc, 1982.
[3] C. S. Smith, Phases and interfaces: an interpretation of microstructure, Trans. AIME **175**, 15-31 (1948).
[4] J. Moon, J. Lee, Ch. Lee, Prediction for the austenite grain size in the presence of growing particles in the weld HAZ of Ti-microalloyed steel, Materials Science and Engineering **A 459**, 40-46 (2007).
[5] Q. Sha, Z. Sun, Grain growth behavior of coarse-grained austenite in a Nb-V-Ti microalloyed steel, Materials Science and Engineering **A 523**, 77-84 (2009).
[6] Q. Yu, Y. Sun, Abnormal growth of austenite grain of low-carbon steel, Materials Science and Engineering **A 420**, 34-38 (2006).
[7] P. R. Rios, Abnormal grain growth development from uniform grain size distributions, Acta Mater. **45**, 4, 1785-1789 (1997).
[8] A. Danon, C. Servant, A. Alamo, J. C. Brachet, Heterogeneous austenite grain growth in 9Cr martensitic steels: influence of the rate and the austenitization temperature, Materials Science and Engineering **A 348**, 122-132 (2003).
[9] S.-J. Lee, Y.-K. Lee, Prediction of austenite grain

growth during austenitization of low alloy steels, Materials and Desing **29**, 1840-1844 (2008).
[10] J. Reiter, C. Bernhard, H. Presslinger, Austenite grain size in the continuous casting process: Metallographic methods and evaluation, Materials Characterization **59**, 737-746 (2008).
[11] M. Dubois, M. Militzer, A. Moreau, J. F. Bussière, A new technique for the quantitative real-time monitoring of austenite grain growth in steel, Scripta Mater. **42**, 867-874 (2000).
[12] J. Fernández, S. Illescas, J. M. Guilemany, Effect of microalloying elements on the austenitic grain growth in a low carbon HSLA steel, Materials Letters **61**, 2389-2392 (2007).
[13] J. C. Bruno, P. R. Rios, The grain size distribution and the detection of abnormal grain growth of austenite in an eutectoid steel containing niobium, Scripta Metallurgica et Materialia **32**, 4, 601-606 (1995).
[14] S. Maropoulos, S. Karagiannis, N. Ridley, The effect of austenitising temperature on prior austenite grain size in a low-alloy steel, Materials Science and Engineering **A 483-484**, 735-739 (2008).
[15] I. Andersen, Ø. Grong, N. Ryum, Analytical modelling of grain growth in metals and alloys in the presence of growing and dissolving precipitates – II. Abnormal grain growth, Acta Metall. Mater. **43**, 7, 2689-2700 (1995).
[16] A. Jacot, M. Rappaz, A combined model for the description of austenitization, homogenization and grain growth in hypoeutectoid Fe-C steel during heating, Acta Mater. **47**, 5, 1645-1651 (1999).
[17] P. R. Rios, M. E. Glicksman, Topological theory of abnormal grain growth, Acta Mater. **54**, 5313-5321 (2006).
[18] M. Shome, Effect of heat-input on austenite grain size in the heat-affected zone of HSLA-100 steel, Materials Science and Engineering **A 445-446**, 454-460 (2007).
[19] M. E. Blanter, Theory of heat treatment, Moscow, Mietallurgija, 1984 (in Russian).
[20] R. Staśko, H. Adrian, A. Adrian, Effect of nitrogen and vanadium on austenite grain growth kinetics of a low alloy steel, Materials Characterization **56**, 340-347 (2006).
[21] H. Adrian, Thermodynamic model for precipitation of carbonitrides in high strength low alloy steels containing up to tree microalloying elements with or without additions of aluminium, Mater. Sci. Tech. **8**, 406-420 (1992).

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