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ON THE M_7C_3 CARBIDES COAGULATION AND COALESCENCE DURING HIGH-TEMPERATURE AUSTENITIZING OF 2% C AND 12% Cr TYPE STEEL

ABSTRACT

The paper presents the results of investigation of carbide grain joints formation during high-temperature austenitizing of steel of about 2% C and 12% Cr type with the additives of tungsten and vanadium. Parameters of distributions of the number of carbide grain joints as the function of austenitizing time at 1150 °C are determined. It was found that in case of carbide grain joints with distinctly outlined boundary division the angles of carbides misorientation forming joints are greater than the angles in joints of the boundary which course is difficult to establish.

Key words: *tool steel, coagulation, carbide grain joints, distribution, diffraction pattern*

INTRODUCTION

The investigation, with the results presented in this paper, is the continuation of studies concerned with coagulation, coalescence and polygonization processes of $(Cr,Fe,W,V)_7C_3$ type carbides during high-temperature austenitizing of steel of about 2% C and 12% Cr type with the additives of W and V, presented in the works [1, 2]. In this part of the studies a special attention is devoted to the determination of parameters of distributions of the number of carbide grain joints as the function of austenitizing time at 1150 °C. The studies of the available literature in the field indicate there have been no complete investigations carried out as yet. It is stated in a few papers only [3-11] that carbide grain joints occur practically in each tool steel hardened; they occur also in these steels under soft annealed state. On the other hand it is known that the driving force to formation of carbide grain joints is their attempt to attain the least energy on the way of diminishing the total boundary surface. The knowledge of this process is essential from the practical point of view because it proceeds also during austenitizing with lower temperatures. Formation of joints and dissolution of smaller carbide grains forming joints occurring with extending of austenitizing time leads to formation of carbide of greater dimensions with a decreased ability to dissolution in austenite.

MATERIAL AND EXPERIMENTAL PROCEDURE

The NCWV/D3 tool steel was the subject of the investigation with the chemical composition presented in Table 1.
The samples for the study were prepared from bars of diameter 13 mm, after plastic working by forging, and soft annealed.

***Table 1.** Chemical composition of the studied NCWV/D3 steel, in wt%*

C	Cr	W	V	Mo	Ni	Mn	Si	Cu	P	S	N
1.95	11.56	1.32	0.31	0.05	0.122	0.44	0.27	0.073	0.024	0.022	0.016

The heat treatment was carried out in an electric furnace with protective atmosphere. The temperature control accuracy was ± 2 ºC. In the experiments, the austenitizing temperature of 1150 °C was assumed for times of 30, 90, 180, and 540 minutes. After the heat treatment, the layer of at least 0.5 mm was removed by grinding to avoid eventually appeared decarburization. An intensive cooling was used during the process of grinding. Metallographic specimens were prepared mechanically using abrasive papers of graininess from 180 to 2500 and afterwards they were polished using aqueous Al_2O_3 suspension. To reveal the carbides, microsections of annealed steel samples after quenching were treated chemically. Dependent on the effect required, to enable counting the joints, the specimens were etched using Murakami's reagent. Metallographic photographs were done by means of Epityp 2 and Neophot 2 microscopes using for this purpose a HI 100 immersion objective and immersion oil of $n_{D(20^\circ C)} = 1.515$.

Counting of the number of carbide grain joints was performed by means of microscope Epityp 2, taking advantage of a rectangle existing in eyepiece of attachment serving to make metallographic micrographs. The rectangle surface area was 0.0095 mm^2. The boundary of carbide joints/couplings was assumed by a counting method as the number of boundaries was simultaneously the number of joints. The average value of the number of carbide grain joints in steel was determined based on the values obtained from 100 visual fields (for austenitizing temperature τ_A = 540 minutes, 95 visual fields), whereas in the austenite grains, based on 200 visual fields. The rectangle was put on the microscopic picture of the etched specimens by a chance manner whereas the numbers of samples after each austenitizing time of 30, 90, 180, and 540 minutes were not less than 3.
Thin foils were prepared in 2 stages:

- electrolytic thinning using abrasive papers of the grain size of 360-800,
- electrolytic thinning by means of a TUNEPOL of STUERS apparatus. A 10 per cent solution of $HClO_4$ in C_2H_5OH was used for thinning at –5 °C and the voltage of 40 V.

Photographs of carbides in thin foil and electronic diffractions were made on BS 613 electron microscope with the accelerating voltage of 100 kV.

STUDY RESULTS AND DISCUSSION

Two methods were used to carry out the indentification of carbides occurring in NCWV/D3 steel hardened after austenitizing at 1150 °C for 90 minutes: (i) on X-ray diffractometer DRON-1.5 taking advantage of carbide extracted particles obtained by means of electrolytic extraction method using MoKα radiation [1], and (ii) on transmission electron microscope BS 613 taking advantage of thin foils. The investigations carried out revealed occurrence only M_7C_3 type carbides in the steel [1, 2] (see Figs. 4, 5).
On presenting kinetics of carbides dissolution during austenitizing NCWV/D3 steel it has been stated [1, 2] that in this steel, *alike in other tool steels*, the carbides of bigger dimensions which lie close to each other, due to the occurrence of diffusion exchange of elements between the neighbouring carbides, have increased their dimensions. Favoured are specifically the carbides lying in the boundaries of austenite grains which increase their dimensions accordingly to the

direction of austenite grains boundaries course (Figs. 1). At a relatively long time of austenitizing a contact point between the carbides may be noticed with the following formation of a boundary between the carbides. The characteristic feature is that these boundaries are rectilinear (Figs. 1b,d, 4).

The number of adhesions/joints appearing in steel undergoes steady changes and is dependent on the temperature and time of austenitizing. A special attention should be paid to the austenitizing at 1150 ºC as the microstructure of the hardened steel is characteristic with the occurrence of high amount of adhesions/joints in the form of chainy conglomerates in the boundaries of austenite grains simultaneously with their small numbers in austenite grains. Some examples of that kind of conglomerates, after etching of specimens by a reagent based on picric acid (Vilella's reagent) and electrolytic etching using 10% aqueous NH_3 solution, in order to reveal the joint boundaries in detail, are presented in Fig. 1a-d.

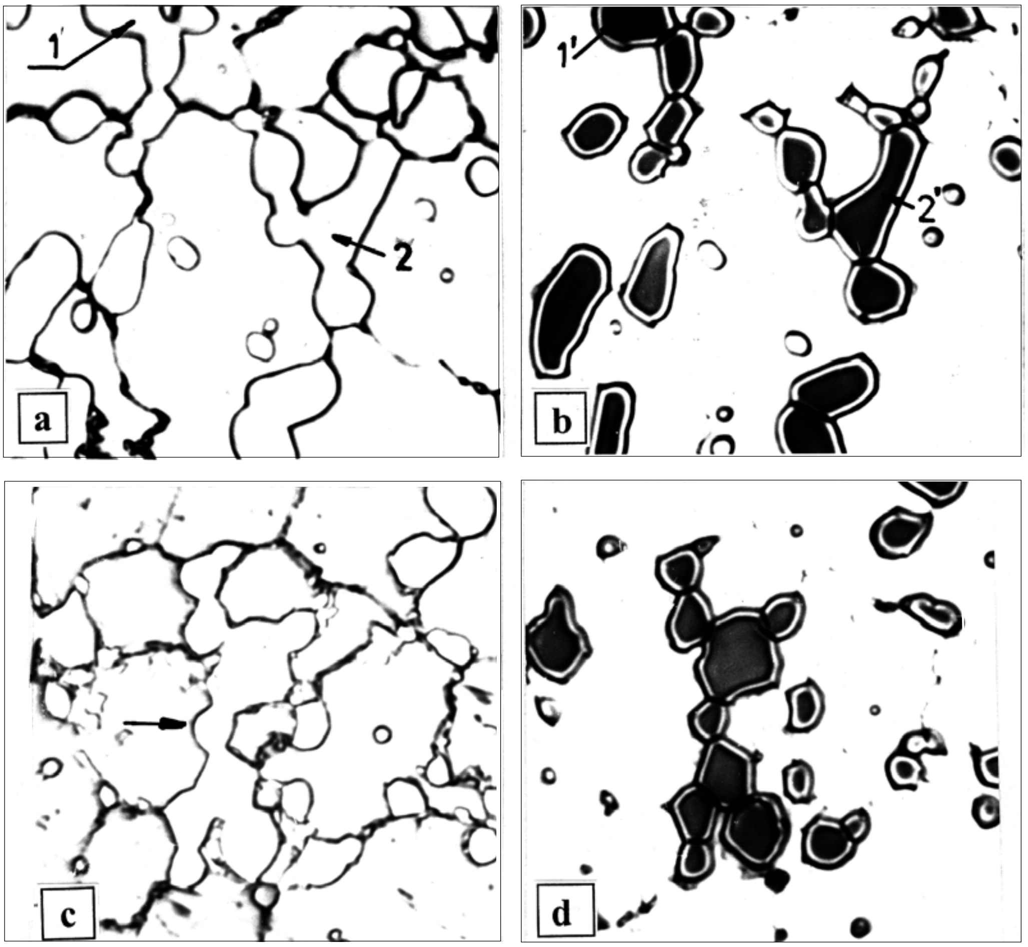

Fig. 1. Pictures of M_7C_3 carbide grain joints and their conglomerates existent in NCWV/D3 steel hardened after austenitizing at 1150 ºC for 90 minutes; magnification 2000 times: (a, c) etched by Vilella's reagent, (b, d) etched electrolytically using 10% aqueous NH_3 solution, voltage applied 12 V, etching time 2 to 3 s, room temperature

Histograms of distribution of the number of carbide grain joints in NCWV/D3 steel hardened after austenitizing at 1150 °C for the periods of 30, 90, 180, and 540 minutes are presented in Fig. 2a-d, with parameters given in Table 2.

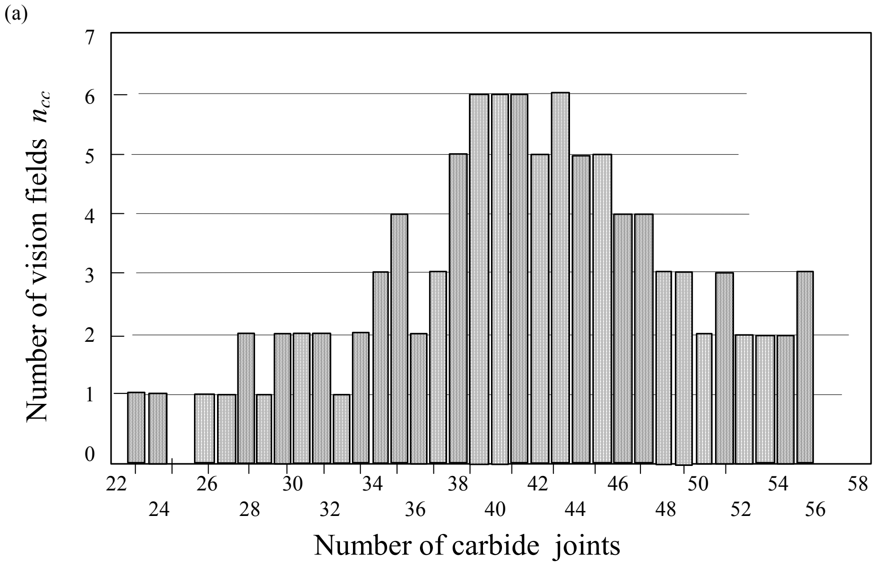

Fig. 2a. Histogram of the number of carbide joints in steel; austenitizing parameters: T = 1150 °C, t = 0.5 h

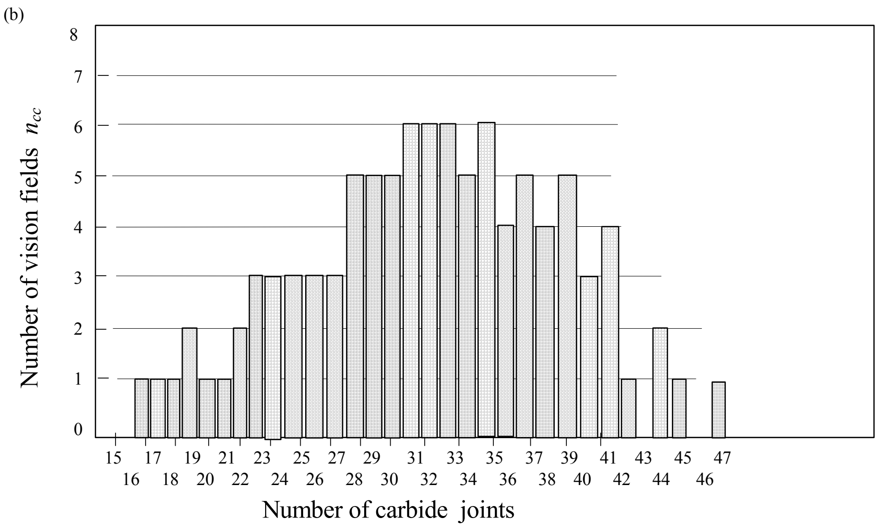

Fig. 2b. Histogram of the number of carbide joints in steel; austenitizing parameters: T = 1150 °C, t = 1.5 h

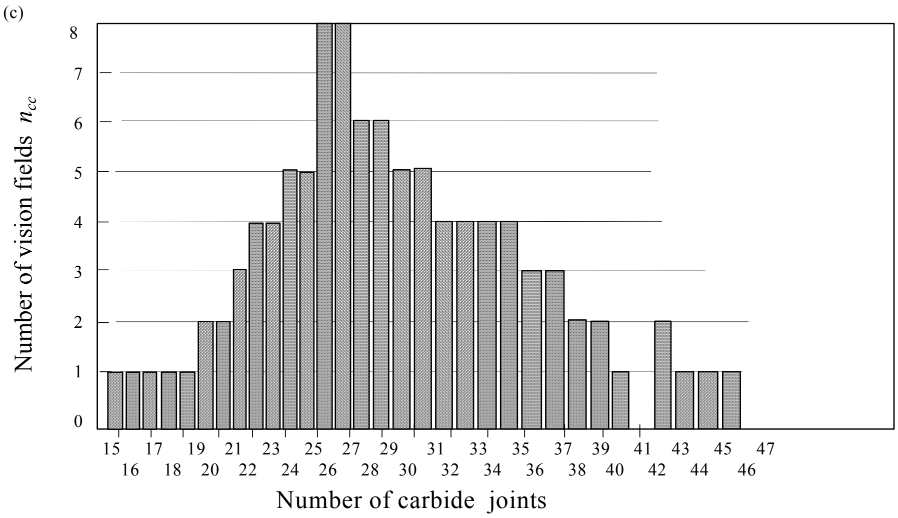

Fig. 2c. Histogram of the number of carbide joints in steel; austenitizing parameters: T = 1150 °C, t = 3.0 h

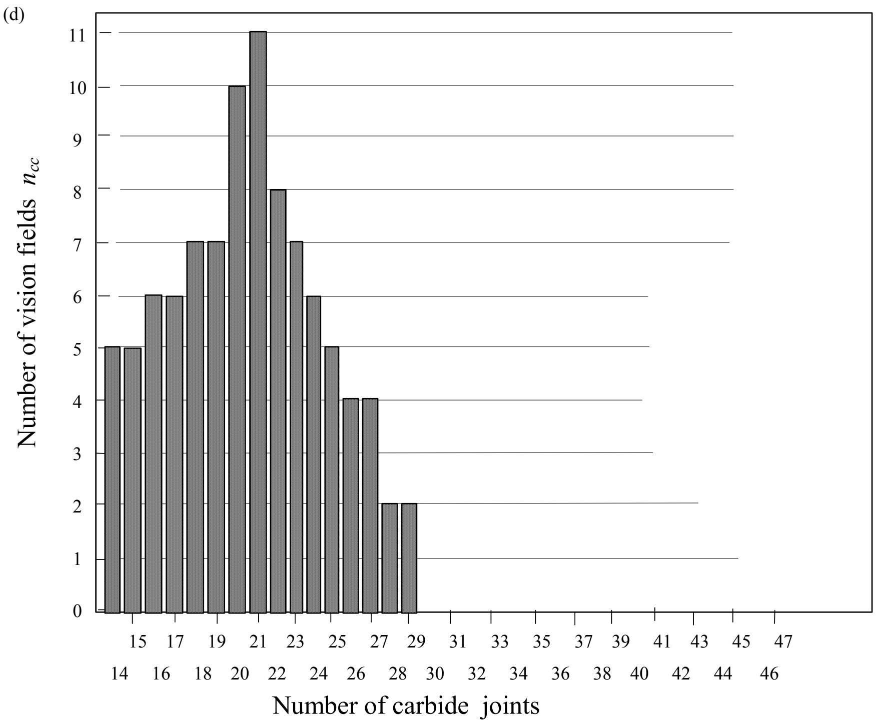

Fig 2d. Histogram of the number of carbide joints in steel; austenitizing parameters: T = 1150 °C, t = 9 h

Table 2. *Parameters of distribution of carbide grain joints in NCWV/D3 steel hardened after austenitizing at 1150 °C for the periods of 30, 90, 180, and 540 minutes*

Variable/Time	30 minutes	90 minutes	180 minutes	540 minutes
Sample size	100	100	100	95
Average	41.03	32.09	28.8	20.6842
Median	41	32	28	21
Mode	40	32	26	21
Geometric mean	40.2665	31.3109	28.0452	20.3227
Variance	58.4334	46.6484	42.5859	14.9205
Standard deviation	7.64418	6.82996	6.52578	3.86271
Standard error	0.764418	0.682996	0.652578	0.396305
Minimum	22	16	14	14
Maximum	55	47	45	29

It results from the data presented in Table 2 and Fig. 2 that distributions of the carbide grain joint number after austenitizing at 1150 °C for the periods of 30, 90, 180 minutes approach a normal distribution. An average number of joints falling into one visual field after austenitizing for 30 minutes equals 41 and decreases with time down to about 21 after austenitizing for 540 minutes. The reason of that phenomenon is the process of dissolution of smaller carbide grains in conglomerates and/or in single joints, what in turn leads to the growth of size of bigger carbides, and in case of single joints, to their disappearance (spheroidization or polygonal carbide shape formation occurs).
With the increase of austenitizing time at 1150 °C the number of joints occurring in austenite grains is also decreasing with a variety of geometric shapes. Typical geometric shapes of carbide grain joints occurring in austenite grains are presented in Fig. 1, and their parameters distribution after austenitizing of the tool steel for the periods of 30, 180, and 540 minutes are given in Table 3.

Table 3. *Parameters of distribution of carbide grain joints in austenite of NCWV/D3 steel hardened after austenitizing at 1150 °C for the periods of 30, 180, and 540 minutes*

Variable/Time	30 minutes	180 minutes	540 minutes
Sample size	200	200	200
Average	7.63	2.49	1.955
Median	8	2	2
Mode	8	1	1
Geometric mean	0	0	0
Variance	12.3649	3.61799	2.41505
Standard deviation	3.51638	1.9021	1.55404
Standard error	0.248646	0.134499	0.109887
Minimum	0	0	0
Maximum	18	9	8

It results from the analysis carried out (Table 3) that the distribution of the carbide grain joint number in the austenite grains of NCWV/D3 steel hardened after austenitizing at 1150 °C for the period of 30 minutes approaches a normal distribution, whereas for the periods of 180 and 540 minutes, a Poisson's distribution [2].

The average number of joints decreases with the increase of austenitizing time at 1150 °C from 7.6 per one visual field after treatment for 30 minutes down to about 2 per one visual field after treatment for 540 minutes (Table 3, Fig. 3).

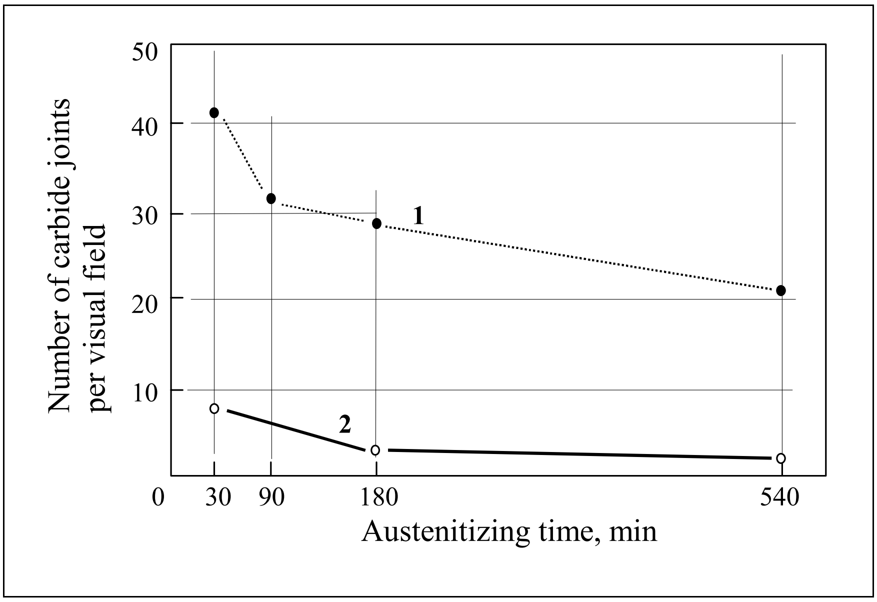

Fig. 3. Effect of austenitizing time on the number of carbide joints 1 – in steel (in austenite grains and baundaries), 2 – in austenite grains

Observations of a number of carbide grains by means of transmission electron microscope on thin foils prepared from the samples of NCWV/D3 steel hardened after austenitizing at 1150 °C revealed that with the extending of austenitizing time the division boundaries in many joints assumed rectilinear shape (see Figs. 4). In the following stage the polygonal shape is assumed by carbides forming joint.

Presented in Figs. 1b,d, and 4 the effect of the rectilinear boundary occurrence in the carbide grains joint does not mean that after a 90-minute austenitizing the boundaries are rectilinear in all joints. An example of joint with a distinctly visible irregular course is shown in Fig. 5.

The studies carried out, concerning crystallographic orientation of carbides forming joints, with the results published in the works [1,2] as well as the present results indicate that their orientation is fortuitous/chance, however, extending of austenitizing time favours crystallographic fitting/matching of carbides. In the investigated joints of carbide grains (Figs. 4, 5) in which the boundaries are clearly outlined, the misorientation angles are 70 deg and 68 deg, respectively, whereas in joints in which the boundaries are not clearly outlined, the boundaries are hardly outlined (Fig. 6), the misorientation angles are lesser and equal 45 deg and 40 deg [2, 1], respectively.

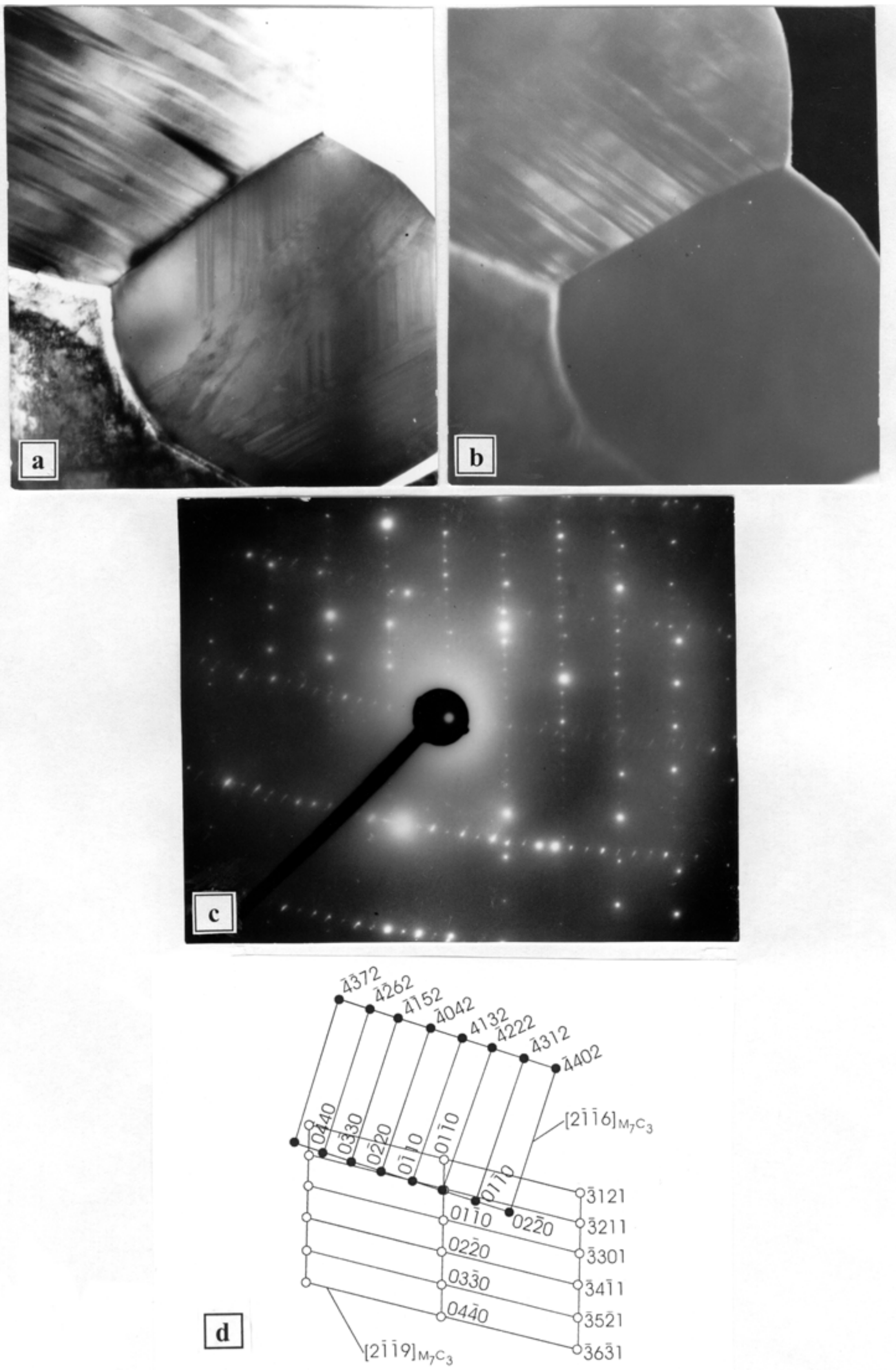

Fig. 4. Joint of two carbide grains with a distinctly outlined rectilinear division boundary. Magnification 20000 times, (a) picture in light field, (b) image in dark field from the reflex (0330), (c) diffraction pattern of two-carbide joint presented in pictures (a,b), (d) solution of the diffraction pattern (c). The angle between the zone axis [2 11 6] of carbide 1 and [2 11 9] of carbide 2 equals 11.2 deg. The angle of total misorientation equals 70 deg

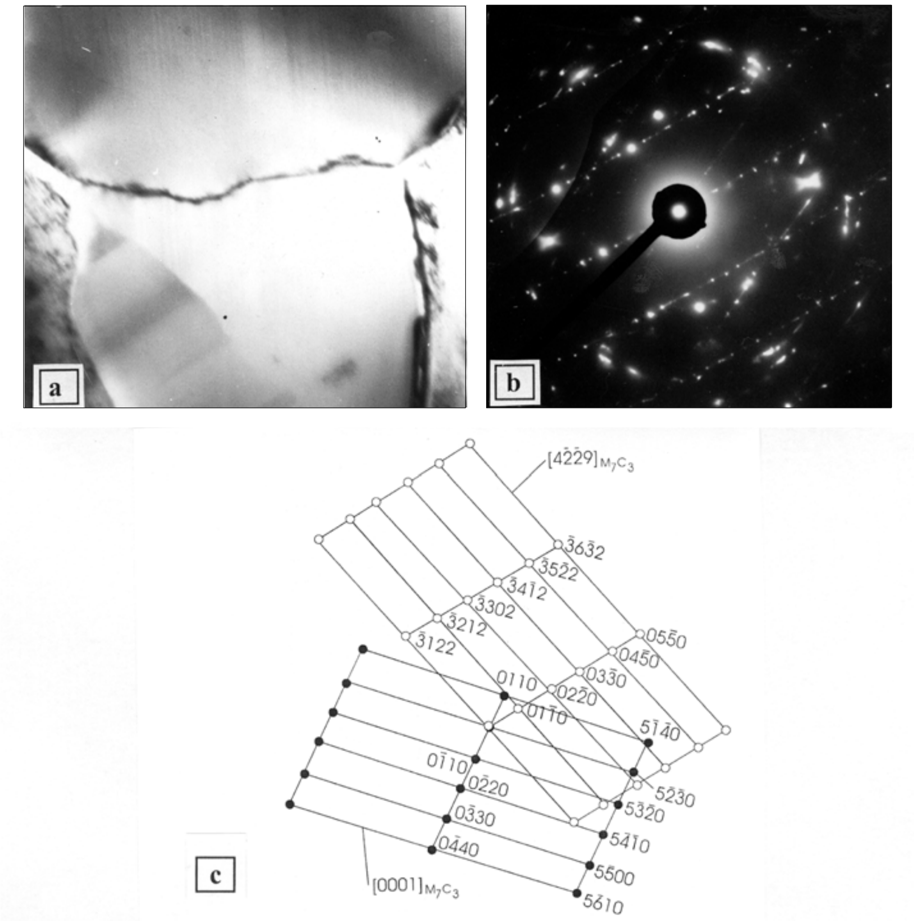

Fig. 5. Joint of two carbides (a) with distinctly irregular boundary division. Thin foil, magnification 13500 times, (b) diffraction pattern of the joint, (c) solution of the diffraction pattern presented in image (a). The angle between the zone axis [0001] and [4 22 9] equals 65 deg, the angle of total misorientation equals 68 deg

Fig. 6. Carbide grain joints (a, b) with hardly outlined grain division. Thin foil, (a) magnification 30000 times, the angle of total misorientation equals 45 deg, (b) magnification 20000 times, the angle of total misorientation equals 40 deg

CONCLUSIONS

The following conclusion may be drawn based on the results obtained with the presented studies:

(1) During a high-temperature austenitizing of NCWV/D3 steel, equaling 1150 °C, the M_7C_3 carbide grain joints are formed arising mainly in the boundaries of austenite grains. The total number of joints in the steel is decreasing with the increase of austenitizing time from 41 after 30 minutes down to about 20/0.0056 mm^2 after austenitizing for 540 minutes time. The distributions of the joint numbers after

austenitizing for 30, 90, and 180 minutes are close to the normal distributions, whereas after 540 minutes, to the Poisson's distribution.

(2) Number of joints arising/forming in austenite grains is distinctly lesser than the number of joints forming in the boundaries of austenite grains and similarly it is decreasing with the austenitizing time from 8, after 30 minutes time, down to about 2 after 540 minutes of the treatment.

(3) Carbide grain joints with distinctly outlined grain division are characteristic with significantly higher values of crystallographic misorientation angle alike in joints of hardly outlined grain division. The angle values of misorientation of some exemplary presented joints are: (i) in joints of distinctly outlined grain division, 70 deg and 68 deg, and (ii) in joints of hardly outlined grain division, 45 deg and 40 deg, respectively.

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