JMEPEG (2003) 12:696-700

Structure and Properties of Nitrocarburized Diffusion Layers Generated on High-Speed Steels

Tomasz Babul, Natalia Kucharieva, Aleksander Nakonieczny, and Jan Senatorski

(Submitted 14 April 2003)

This work analyzes the structure and properties of nitrocarburized diffusion cases generated on M2 type high-speed and 321 stainless steels in a thermochemical. Application of variable process temperatures in the range of 450-600 °C and a variable process duration (2-6 h) enabled observation of growth kinetics of the layers on tested steel grades. Evaluation of properties of the cases obtained comprised hardness measurements and wear tests, carried out by the 3 cylinder-cone method. The evaluation showed that the nitrocarburizing process developed for high-speed and stainless steels yields hard surface layers with beneficial functional properties.

Keywords hardness, heat treatment, high-speed steel, nitrocarburized case, stainless steel, wear resistance

1. Introduction

The solution of durability and operational reliability problems of machine components, technological instrumentation, and tooling is connected, in most cases, with the production and utilization of high-alloy steel and with optimization of its heat-treating conditions. Frequently, these operations are not sufficient and it is necessary to search for other solutions. Such solutions obviously include surface treatment methods, which ensure that layers are obtained with the required properties. Their range covers diffusion processes yielding nitrocarburized layers in final thermochemical treatment processes of machine elements, tooling, and technological instrumentation. The generation of nitrocarburized layers on working surfaces by such methods makes possible the extension of their normal service life by three to seven times.[1,2,3,4]

Due to simplicity and operational safety, easy waste disposal, and the possibility of process control ensuring the formation of layers with predicted chemical and phase structure, thermochemical processes using activated powder packs seem to have several advantages.

2. Materials and Experimental Procedure

The main objective of the present work was to define the kinetics of the structure and phase composition, to perform microhardness measurements, and to determine the frictional wear resistance of nitrocarburized layers formed on M2 and 321 steels. The chemical composition of these steels is given in Table 1.

Nitrocarburizing processes were carried out at temperatures of 450-600 °C in durations of up to 6 h using powder packs based on carbon and potassium ferrocyanide. For homogenization of M2 specimen structure, prior to thermochemical treating, a typical heat treatment process was applied; i.e., oil quenching from a temperature of 1220 °C followed by a triple temper at 550 °C. Nitrocarburizing of 321 stainless was carried out without prior heat treatment.

Tomasz Babul, Aleksander Nakonieczny, and **Jan Senatorski,** Institute of Precision Mechanics, Warsaw, Poland; and **Natalia Kucharieva,** National Technical University, Minsk, Belarus. Contact e-mail: tomasz@imp.edu.pl.

Table 1 Chemical Composition of Tested Steels

	Chemical Composition %							
Grade of Steel	C	Cr	Ni	Ti	W	Mo	V	Fe
M2	0.85	4.0	…	…	6.0	5.0	2.0	Balance
321	0.10	17-19	10	1	…	…	…	Balance

Metallurgical evaluation was carried out with an optical microscope and a hardness tester. For structural analysis, x-ray diffraction (XRD) was used in conjunction with metallographic tools. This analysis included:

- metallographic observations using the Neophot 21 light microscope (Carl Zeiss, Jena, Germany), with magnifications from 100-500 X;
- phase composition, determined by x-ray structural analysis using the DRON-3 diffractometer with counter feed rate of 1°/min, and CuK_{α} radiation;
- hardness measurements using the PMT-3 microhardness tester (OAO "LOMO", Saint Petersburg, Russia) (applied load – 0.49 N, indentation time – 10 s); and
- micro XRD analysis using the Cameca analyzer (Cameca, Courbevoie, France), with the application 18 kV and current dependent on tested element.

Wear tests were carried out by the 3 cylinder-cone method[5] with a slide velocity of 0.58 m/s and unit loads of 50 and 400 MPa. Total wear test time was 100 min, which corresponded to a slide path of 3470 m. Wear depth was measured microscopically every 10 min. Mixed friction conditions were maintained using drip-feed lubrication with Lux-10 oil (IMP, Warsaw, Poland) (rate of 10 drops/min).

3. Results

The temperature/time dependence testing of diffusion case thickness obtained on M2 and 321 steels showed that the case

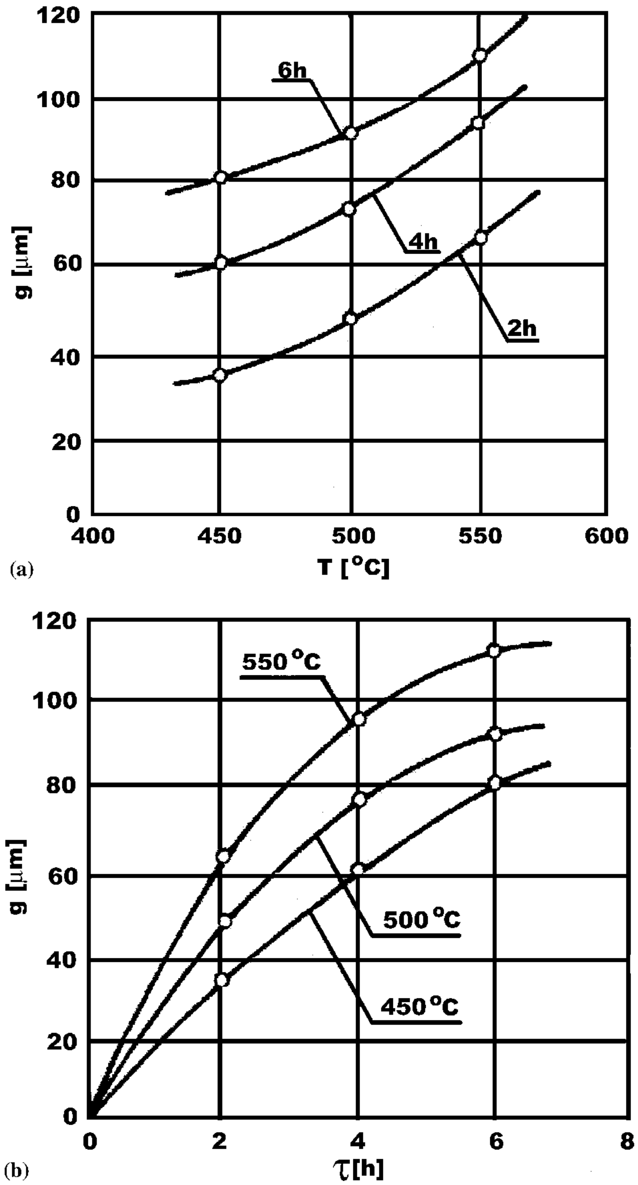

Fig. 1 Nitrocarburized case depth on M2 steel vs **(a)** process temperature; **(b)** process duration

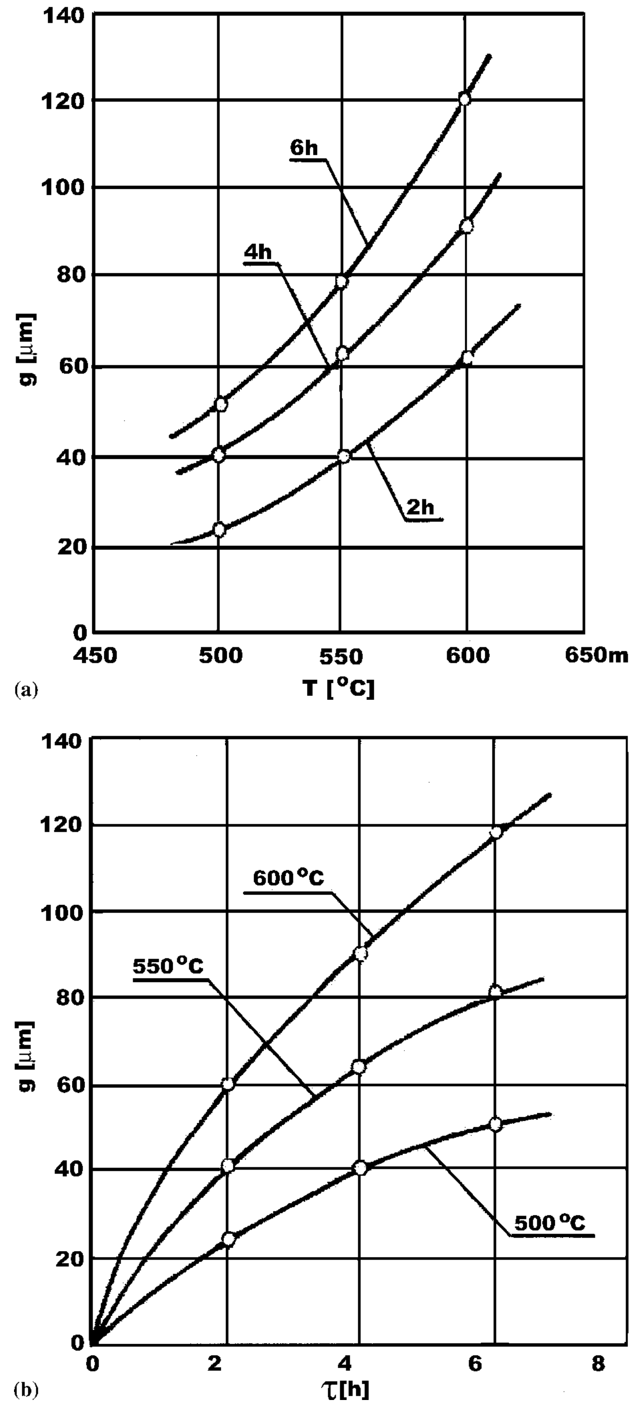

Fig. 2 Nitrocarburized case depth on 321 stainless steel versus **(a)** process temperature; **(b)** process duration

depth variation has an exponential correlation with process temperature and a parabolic one with process duration (Fig. 1 and 2).

Metallographic observations showed that the nitrocarburized case on 321 steel, treated at 500, 550, and 600 °C (duration 6 h) is composed of three zones:

1) A thin, discontinuous hematite zone. This zone grows and increases its discontinuity with temperature growth. It is characterized by low hardness of 160-270 HV 0.05. This is an undesirable zone in layer structure.
2) A discontinuous, heterogeneous zone consisting mainly of an oxycarbonitride $(Fe,Cr,Ni,Ti)_3(NCO)_4$ with a spinel structure and solid solution γ. This zone is built by near-cube grains, which are well interconnected, fine grained, and high alloyed. They are hard and feature high strength and corrosion resistance. The γ phase does not occur in the surface part of this zone, but its content grows with distance from surface. However, in this part an increased content of C and N is observed. Measured microhardness of this zone decreases with depth from 1150 to 950 HV (Fig. 3).
3) A homogeneous zone of solid solution γ, characterized by

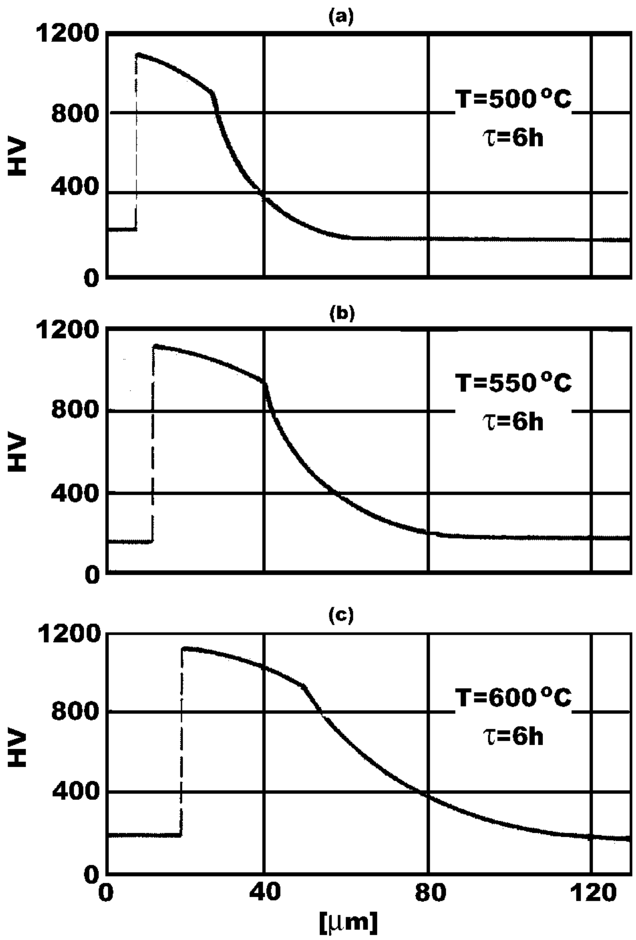

Fig. 3 Microhardness distribution in the nitrocarburized case cross section on 321 steel for three process temperatures: **(a)** 500 °C; **(b)** 550 °C; **(c)** 600 °C (process duration 6 h for all)

good ductility and impact strength with relatively high hardness and strength. Nitrogen and particularly carbon content decrease with distance from surface. In the same way the zone microhardness drops from 950 to 200 HV, where the last figure corresponds to the microhardness of the core.

The just-described three zones of the nitrocarburized case on 321 steel differ distinctly from one another by individual structures and clear-cut boundaries. At the same time, a continuous change of microhardness is observed within each zone and simultaneously, a sharp change in the character of microhardness distribution and its value from one zone to the next. However, the differences in microhardness between zones are rather small and do not cause any deterioration of good functional properties of nitrocarburized cases and especially of its good wear resistance.

Micro XRD analysis showed a nonuniform distribution of elements (Fe, Cr, Ni, Ti) in the heterogeneous zone on 321 steel and a uniform one in the solid solution zone located below.

In the case of nitrocarburized M2 steel, the structural, chemical, and phase changes occur in its surface zones. A thin

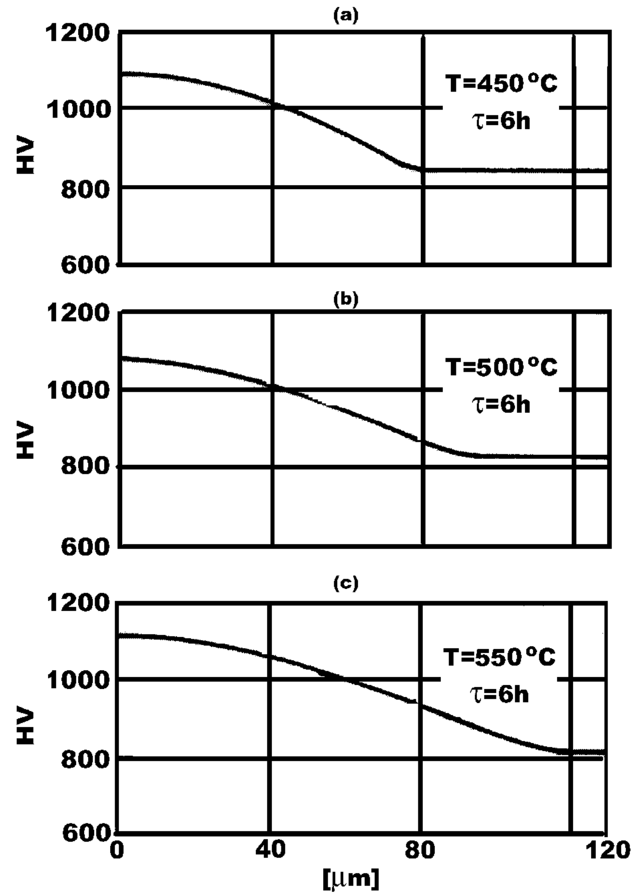

Fig. 4 Microhardness distribution in the nitrocarburized case on M2 steel for three process temperatures: **(a)** 450 °C; **(b)** 500 °C; **(c)** 550 °C (process duration 6 h)

oxycarbonitride layer is formed on the surface and its thickness grows from 1-4 μm with the rise of process temperature from 450 °C up to 550 °C. Below this zone, the main heterogeneous zone is extended, composed of the α-phase, MoC, WC, VC, Fe_3C carbide inclusions, CrN, Cr_2N nitride inclusions, and the area characterized by a dominant presence of the $(Fe,Cr,Mo,W)_3CN$ type carbonitride of the ε phase. The ε carbonitride, which forms the main phase present in the diffusion case on high-speed steel, changes its composition with distance from the surface. It is easily strengthened by alloying elements. Its suitable connection with the bcc lattice of the α-phase and with high coherence with carbonitride inclusions of alloying elements (of MoCN, WCN type) ensures high hardness and good wear resistance of the nitrocarburized layer. An additional strengthening phase of the layer on M2 steel in thermochemical treatment processes carried out in a powder pack is the $Fe_4[Fe(CN)_6]_3$ type carbonitride. In comparison with the main carbonitride ε, it is much richer in carbon and nitrogen. X-ray testing at various layer depths shows that the highest density of $Fe_4[Fe(CN)_6]_3$ type carbonitride occurs in the surface part of the obtained layers.

Microhardness of the nitrocarburized case on M2 steel is shown in Fig. 4. It varies continuously across the entire layer from 1145 HV down to 830 HV, i.e., to the hardness of the core. Such high microhardness values are observed when the

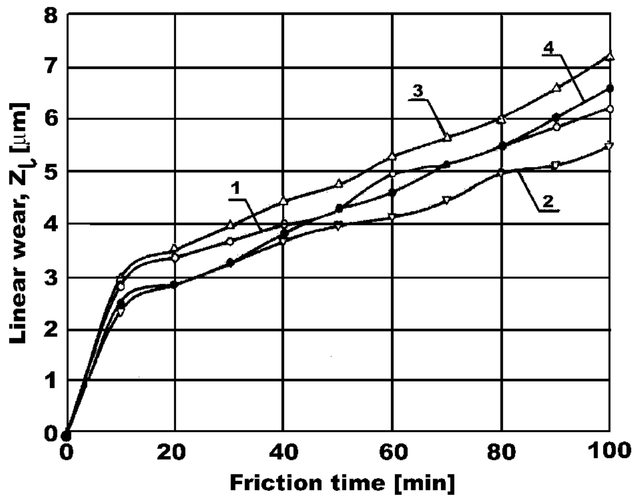

Fig. 5 Linear wear vs sliding time for nitrocarburized M2 steel determined with a unit load of 50 MPa for three process temperatures: (1) 450 °C; h, (2) 500 °C; (3) 550 °C; and (4) no surface treatment (process duration 6 h at all temperatures)

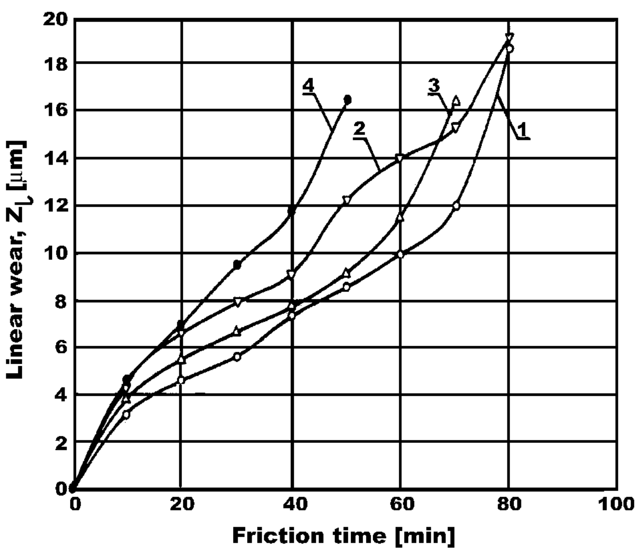

Fig. 6 Linear wear vs sliding time for nitrocarburized M2 steel, determined with unit load of 400 MPa for three process temperatures: (1) 450 °C; (2) 500 °C; (3) 600 °C; and (4) no surface treatment (process duration 6 h at all temperatures)

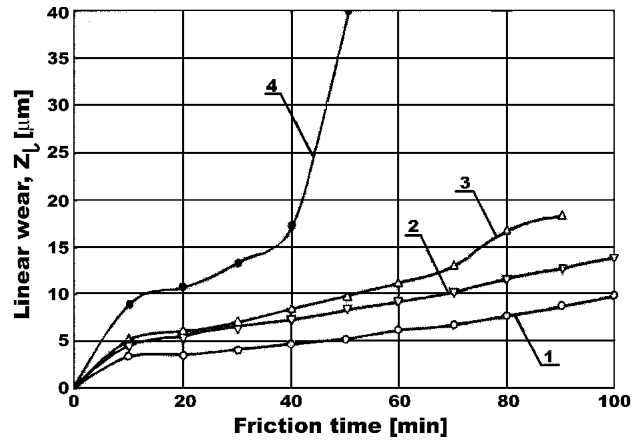

Fig. 7 Linear wear vs sliding time for nitrocarburized 321 steel, determined with a unit load of 50 MPa for three process temperatures: (1) 500 °C; (2) 550 °C; (3) 600 °C; and (4) no surface treatment (process duration 6 h at all temperatures)

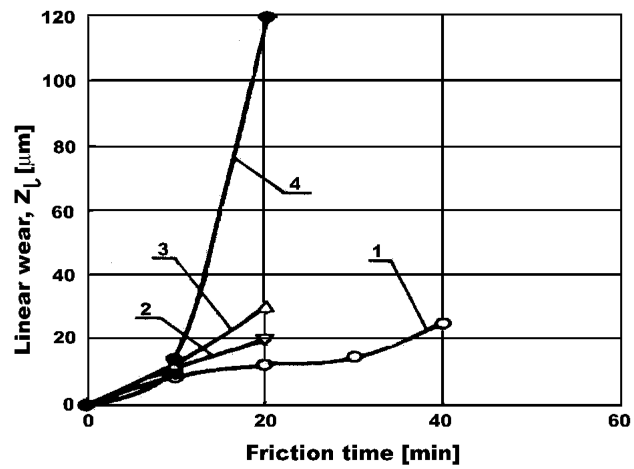

Fig. 8 Linear wear vs sliding time for nitrocarburized 321 steel, determined with a unit load of 400 MPa for 3 process temperatures: (1) 500 °C; (2) 550 °C; (3) 600 °C; and (4) no surface treatment (process duration 6 h at all temperatures)

inclusion phases produce totally coherent structures with the α-phase,[6] which was obtained on M2 steel, nitrocarburized at all process temperatures. Metallographic observations revealed no distinct boundary between layer and substrate.

Case depths were determined by microhardness traverses across the layer down to the core. It was found that the character of microhardness profile in nitrocarburized cases on the steel is not dependent on thermochemical treatment process temperature or time.

Tribological properties of the tested steels are presented by plots in Fig. 5, 6, 7, and 8 showing linear wear variation versus sliding time. Figure 5 and 6 show four treatment versions of M2 steel, tested with a unit load of 50 MPa and 400 MPa, while Fig. 7 and 8 show corresponding curves for 321 steel, tested under the same loading conditions. A comparison of results shows a strong influence of the unit load on the wear curves of both steel grades. Of the tested material and treatment versions of M2 steel, the highest wear resistance was exhibited under both unit loads by that steel when nitrocarburized at 450 °C. On the other hand, for 321 steel, all nitrocarburizing versions enhanced tribological properties in comparison with the untreated condition.

4. Summary

Results show that powder-pack nitrocarburizing processes developed for high-speed and stainless steels enabled the formation of diffusion layers of high hardness, featuring very favorable functional properties, confirmed by wear tests.

Experimental data obtained on some types of tools and technological instrumentation subjected to the nitrocarburizing process, as described in this paper, confirmed its favorable effect on the durability of machining, cold working, and hot working tools.

Acknowledgment

This work was supported by a grant from the State Committee for Scientific Research under the auspices of a research cooperation agreement between Poland and Belarus.

References

1. D.A. Prokoškin: "Thermochemical Treatment of Metals—Carbonitridin," Moscow *Metallurgija, Mašinostrojenije*, 1984, p. 240 (in Russian).
2. V.M. Zintchenko and V.Y. Syropiatov: "New Abilities of the Gas Nitriding as a Method for Anticorrosive Treatment of the Machinery Parts," 11th Cong. Int. Fed. Heat Treatment and Surface Eng. and 4th ASM Heat Treatment and Surface Eng. Conf. Eur., 19-21 October, 1998: Proc. Vol. 3, Milano, 1998, pp. 9-17.
3. Michel Gantois: "Carburizing and Carbonitriding Procedures of Steels," Switzerland Patent 688801, MPKO S23S 008/22 Solo Foues. Ind. S.A.—Nr 02168/94. Patent application 07.07.94, publ. 31.03.98.
4. B. Edenhofer, W. Lerche, and W. Göhring: "Gaseous Nitrocarburising— Problems and Solutions with Today Process Technology," *Heat Treatment of Metals*, 1995, *22*(12), pp. 27-33.
5. PN-83/H-04302 "Friction Test in 3 Cylinder-Cone" (in Polish).
6. J.N. Lachtin and J.D. Kogan: "Carbonitriding of Steel," Moscow Mašinostrojenije, 1976, p. 220 (in Russian).