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Effects of heat treatment on the microstructures and mechanical properties of a new type of nitrogen-containing die steel

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Abstract: Nitrogen can increase the strength of steels without weakening the toughness and improve the corrosion resistance at the same time. Compared with conventional nitrogen-free die steels, a new type of nitrogen-containing die steel was developed with many superior properties, such as high strength, high hardness, and good toughness. This paper focused on the effects of heat treatment on the microstructures and mechanical properties of the new type of nitrogen-containing die steel, which were investigated by the optimized deformation process and heat treatment. Isothermal spheroidal annealing and high-temperature quenching as well as high-temperature tempering were applied in the experiment by means of an orthogonal method after the steel was multiply forged. The mechanical properties of nitrogen-containing die steel forgings are better than the standard of NADCA #207-2003.

Keywords: die steels; nitrogen; heat treatment; microstructure; mechanical properties; forgings

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1. Introduction

In recent years, with the development of manufacturing industry and the appearance of new materials, the working conditions of tools and dies become more and more severe. Thus, new requirements are proposed on die steel's performances, qualities, and varieties. Nitrogen can increase the strength of steels without weakening the toughness and improve the corrosion resistance at the same time. According to these characteristics, high-nitrogen steels, especially high-nitrogen austenitic stainless steels, have been researched intensively in recent years [1-3]. However, the effects of nitrogen element on the microstructures and mechanical properties of die steels have not been investigated until now.

H13 steel is a typical hot-working die steel which is widely used for hot extrusion dies, forging dies, and casting dies [4-5]. A new type of nitrogen-containing die steel based on H13 steel was applied for the patent in 2009 [6]. In this paper, the effects of heat treatment on the microstructures and mechanical properties of the nitrogen-containing die steel were investigated. The phase transformation temperatures obtained in the thermal expansion experiment provided a significant reference for the heat treatment process. The as-annealed tested steels were quenched and tempered by means of orthogonal test, and then the mechanical properties were tested under different microstructural conditions.

2. Experimental

2.1. Materials

The nitrogen-containing tested steel was primarily smelted in a 50-kg vacuum induction melting furnace and

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then refined in an electro-slag-remelting furnace. The refined steel was preheated at 850°C for 3 h, subsequently heated to 1200°C, and held for 2 h; then multiple forging was carried out for grain refinement and mechanical propery improvement [7]. The chemical composition of the tested nitrogen-containing steel is listed in Table 1.

Table 1. Chemical composition of the tested steel wt%

C	Si	Mn	P	S	Cr	Mo	V	N	Fe
0.35-0.40	0.80-1.10	0.40-0.50	≤0.025	≤0.005	4.90-5.50	1.40-1.70	0.80-1.10	0.025-0.045	Bal.

2.2. Methods

The tested steel was machined to the dimensions of ϕ4 mm×10 mm to measure the phase transformation temperatures. The measurement experiment was conducted on a Baehr DIL805A thermal dilatometer under vacuum condition to prevent the surface oxidation of samples. The measured temperature region was from room temperature to 1030°C.

The heat treatment of the nitrogen-containing tested steel, which included annealing, quenching, and tempering, was conducted in a chamber electric furnace. The hardness at annealing state was tested on an XHB-3000 Brinell hardness (HBW) tester, and the hardness at quenching and tempering states was measured with a TH320 Rockwell hardness (HRC) tester. Room-temperature tensile tests and room-temperature Charpy V-notched impact tests were conducted on a CMT4105 universal test machine and a JB-30B impact test machine, respectively. Furthermore, the microstructures were observed by means of Leica DM2500M optical microscope and Zeiss EVO18 scanning electron microscope (SEM).

3. Results and discussion

3.1. Phase transformation temperatures of the tested steel

The phase transformation temperatures of the nitrogen-containing die steel, including the lower critical temperature of austenitic transformation (Ac_1), the upper critical temperature of austenitic transformation (Ac_3), the starting temperature of martensitic transformation (Ms), and the finishing temperature of martensitic transformation (Mf), are the primary references of heat treatment. According to Ref. [8] about the steel determination of critical point-dilatometric methods and the working conditions of die steels, the austenitizing temperature of the tested steel was 1030°C. To obtain various phase transformation temperatures at the same time, the experimental procedures were decided as follows: first, the sample was heated to 500°C at a rate of 10°C/s, subsequently heated to the austenitizing temperature of 1030°C at a rate of 200°C/h, and held for 15 min at this temperature; then, the sample was cooled to 500°C at a rate of 200°C/h, subsequently to the ambient temperature at a rate of 20°C/s.

The thermal volumetric expansion coefficient of the nitrogen-containing tested steel measured by the above method is shown in Fig. 1. As well-known, the rank of specific volume presents the transformation of martensite, pearlite, and austenite, respectively. In Fig. 1, with the rise of temperature, an approximately linear expansion can be obtained when the temperature is below 800°C. Then, there are two inflection points at 800 and 847°C due to phase transformation. In addition, as an opposite trend, the volume shrinkage is investigated from 800 to 847°C, owing to the fact that the specific volume of austenite is smaller. The sample re-expands when the temperature is above 847°C, implying that the austenitizing process is finished. Thus, the Ac_1 and Ac_3 points of the nitrogen-containing tested steel can be inferred to be 800 and 847°C, respectively.

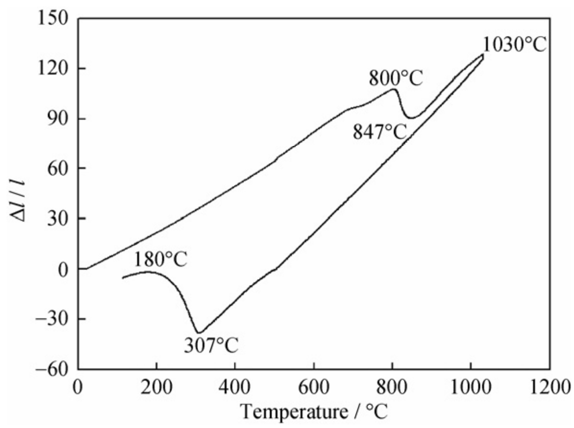

Fig. 1. Thermal expansion curve of the tested steel, where Δ*l* stands for the expanding quantity of line length, $\Delta l/l_0$ the dependent variable, and l_0 the length of the initial sample.

In contrast, the volume shrinkage takes place during sample cooling from 1030°C. When the supercooled austenite induces martensite transformation, the volume expansion occurs, because the specific volume of martensite is bigger than that of austenite. An abrupt change from 307 to 180°C represents the martensite formation process in the tested steel.

The measured phase transformation temperatures are shown in Table 2. Since the chemical composition of the tested steel is similar to that of AISI H13 steel except for nitrogen, the transformation points of AISI H13 steel are also listed in Table 2 as reference. The phase transformation temperatures of the tested steel are distinctly lower than those of AISI H13 steel. It is considered that nitrogen is an important alloying element that can expand the γ-phase field and has the tendency to decrease the austenitic and martensitic transformation temperatures.

Table 2. Comparison of phase transformation temperatures °C

Steel	Ac_1	Ac_3	Ms	Mf
Tested steel	800	847	307	180
AISI H13 steel	860	915	340	215

3.2. Annealing process of the tested steel

Generally, annealing as a pre-heat treatment for die steels should be immediately performed after forging to refine the grain size, reduce the hardness, and improve the machinability [9-10]. Although the tested steel only contains 0.4wt% carbon element, it belongs to the hypereutectoid steel because of over 8wt% alloying elements. Besides, spheroidal pearlite is an appropriate microstructure for machining. Therefore, the pre-heat treatment of the tested nitrogen-containing steel is determined to be isothermal spheroidal annealing, as shown in Fig. 2.

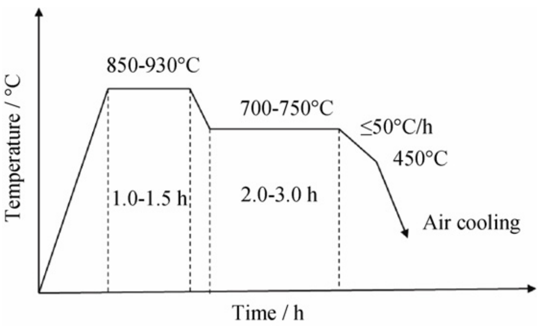

Fig. 2. Schematic diagram of the annealing process in the experiment.

Fig. 3 shows the as-annealed microstructure of the tested nitrogen-containing steel. As can be seen, it essentially consists of a ferritic matrix with a homogeneous distribution of spheroidized carbides. The room-temperature mechanical properties of the as-annealed nitrogen-containing steel are given in Table 3. It is indicated that the as-annealed nitrogen-containing steel achieves a good processing performance, and the hardness meets the standard of NADCA #207-2003 [11], which is specified not to be higher than HBW 235.

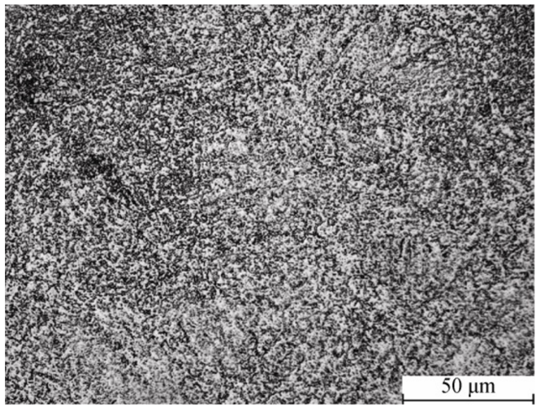

Fig. 3. As-annealed microstructure of the tested steel.

Table 3. Room-temperature mechanical properties of the as-annealed nitrogen-containing steel

R_m / MPa	R_p / MPa	A / %	Z / %	HBW
688.06	320.92	24.00	60.62	200.9

Note: R_m is the tensile strength, R_p the yield strength, A the elongation, and Z the reduction of area.

3.3. Quenching process of the tested steel

To obtain the good comprehensive mechanical properties of strength and toughness, the heat treatment of quenching followed by tempering is needed for hot-working die steels after rough machining. The purpose of high-temperature quenching is to develop high strength and hardness, in company with the improvement of wear resistance [12]. Moreover, high-temperature tempering can eliminate quenching-induced residual stress and retained austenite, so the ductility and toughness can be improved. Therefore, the final heat treatment of the nitrogen-containing die steel was determined to be high-temperature quenching and high-temperature tempering to achieve an optimum combination of mechanical properties.

In the quenching process, the samples of the nitrogen-containing steel were austenitized at 1020°C followed by oil quenching, while the holding time varied from 25 to 90 min. As shown in Fig. 4(a), 30 min is selected as the optimal holding time due to the highest hardness value of HRC 55.5. In addition, Fig. 4(b) shows the hardness of samples austenitized at different temperatures for 30 min. The results show that the hardness is greatly influenced by quenching temperature. As the quenching temperature rises, the solid solubilities of carbon and other alloying elements in quenched martensite increase due to carbides dissolving gradually, so the hardness of the tested steel is improved. Oil cooling after quenching can increase the amount of retained austenite, which is in favor of the structural transformation of tempering. However, if the quenching temperature is too high, there may be the austenite grain growth and the hardness decrease of the tested steel.

As shown in Fig. 5, the as-quenched microstructure of the tested steel consists of martensite and retained austenite as well as some undissolved carbides. More and more carbides dissolve into the matrix with the rise of quenching temperature. At the same time, the grains grow constantly, and serious grain coarsening occurs when the temperature reaches 1200°C. In addition, excessive carbon and other alloying elements dissolve gradually in the austenite matrix at this temperature, leading to the decrease of the Ms temperature and the increase of austenite stability. As a result, the retained austenite increases and the hardness value decreases at room temperature.

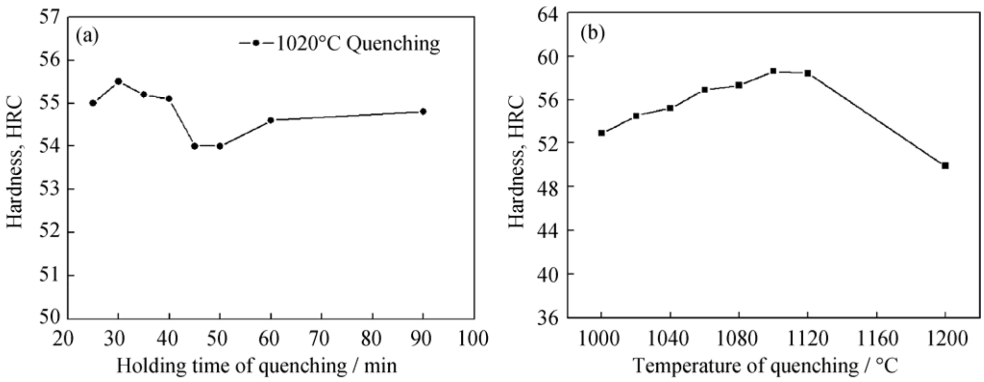

Fig. 4. Hardness of the tested steel after various holding times (a) and at different quenching temperatures (b).

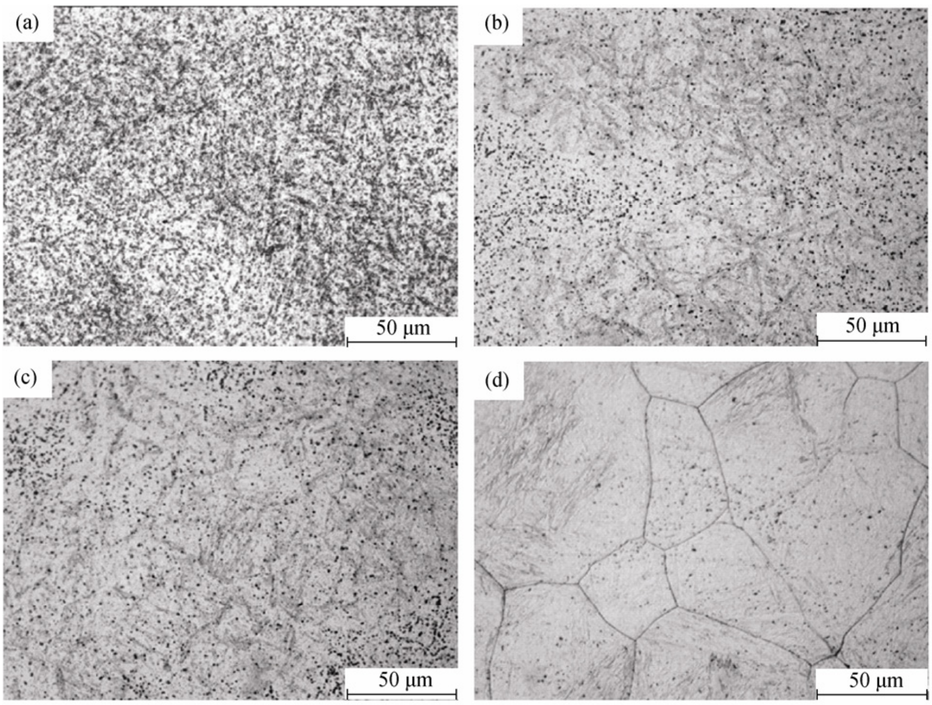

Fig. 5. Microstructures of the tested steel austenitized at different temperatures for 30 min: (a) 1020°C; (b) 1040°C; (c) 1100°C; (d) 1200°C.

3.4. Tempering process of the tested steel

To eliminate residual stress caused by quenching and improve the ductility and toughness of die steels, twice or thrice high-temperature tempering are usually carried out [13-14]. Premium and superior quality H13 steels are mostly subjected to 540-650°C high-temperature tempering to improve the toughness and reduce the temper brittleness, which is caused by the transformation of retained austenite in the die steel [15].

To confirm the effect of tempering temperature on the mechanical properties, the tested steel was austenitized at 1040°C for 30 min, oil quenched, and subsequently tempered at 350, 400, 450, 500, 550, 600, and 650°C for 2 h, respectively. The curves of hardness and impact toughness (α_k) to tempering temperature are shown in Fig. 6. The hardness increases and the impact toughness decreases when the tempering temperature increases from 350 to 500°C. When the sample is tempered at around 500°C, significant secondary hardening occurs and the hardness reaches the peak value, HRC 55.3, of the tested steel. It can be attributed to the enrichment and segregation of carbide forming elements at 500°C, such as V, Mo, and C. As the tempering temperature further increases, the hardness decreases dramatically and drops to HRC 38.3 at 650°C. As for the impact toughness, an opposite trend appears compared with the hardness. The hardness peak of secondary hardening is exactly corresponding to the trough of impact toughness due to temper brittleness. The impact toughness peak, 14.8 J·cm^{-2}, appears at 650°C.

As shown in Fig. 7, the as-tempered microstructure of the tested steel consists of fine tempered sorbite with a few of carbide particles. With the increase of tempering temperature, more and bigger carbides precipitate. At the same time, the tempered sorbite coarsens gradually.

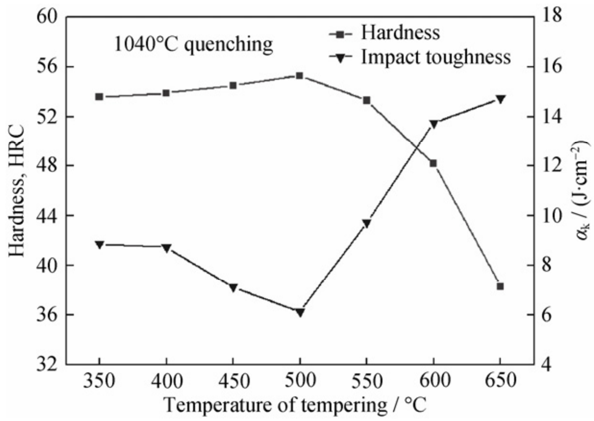

Fig. 6. Hardness and impact toughness of the tested steel at different tempering temperatures after quenched at 1040°C.

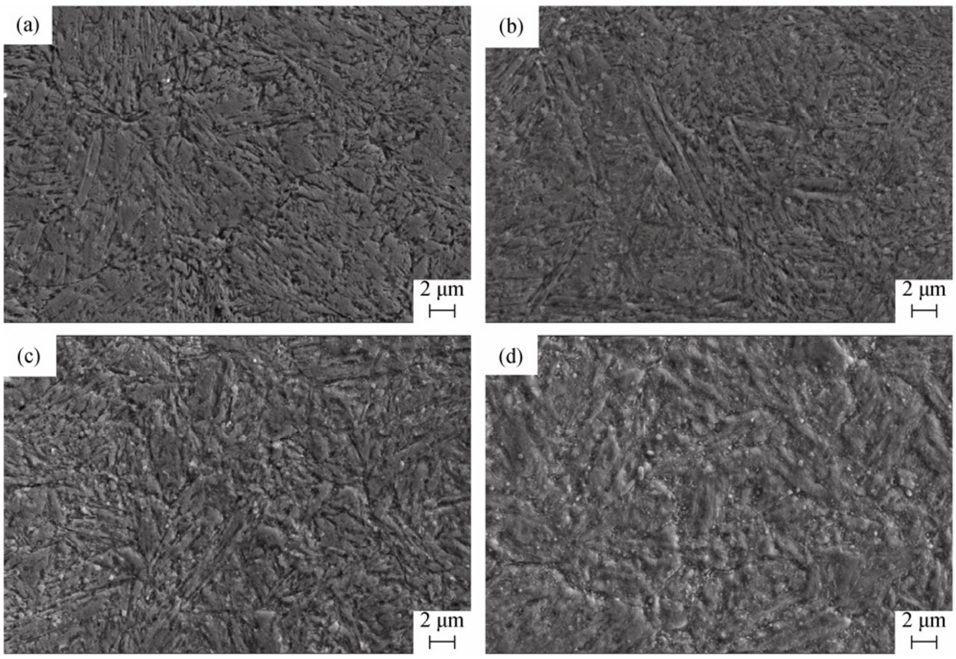

Fig. 7. SEM images of the tested steel quenched at 1040°C when tempered at (a) 500°C, (b) 550°C, (c) 600°C, and (d) 650°C.

3.5. Optimization of mechanical properties

Optimizing the heat treatment parameters of nitrogen-containing die steels can effectively improve the comprehensive mechanical properties and achieve a good balance between strength and toughness, which can significantly contribute to the service life of dies.

In this section, the tested steel was quenched at different temperatures followed by tempering twice. To avoid the appearance of secondary hardening of tempering and reduce the working time of actual heat treatment, oil cooling and air cooling were employed for the first and second tempering, respectively. The obtained mechanical properties of the tested steel are shown in Fig. 8.

As seen in Figs. 8(a) and (b), the yield strength and hardness decrease with the decrease of quenching temperature or the increase of tempering temperature. However, it is notable that all the treatment combinations between quenching and tempering, except for 1000°C quenching and 600°C tempering, present that the yield strength is higher than 1370 MPa and the hardness is higher than HRC 46. It suggests that solid solution nitrogen improves the strength and hardness substantially. Fig. 8(c) shows that when the samples are quenched at the same temperature, the impact toughness essentially increases as the tempering temperature increases. According to the NADCA #207-2003 specification, α_k should be higher than 10 J·cm^{-2} when the hardness is about HRC 46. In this paper, the α_k value of the tested steel is beyond 14 J·cm^{-2}, while the hardness is HRC 49. On the other hand, if the impact toughness keeps 10 J·cm^{-2}, the hardness can reach HRC 46-54.

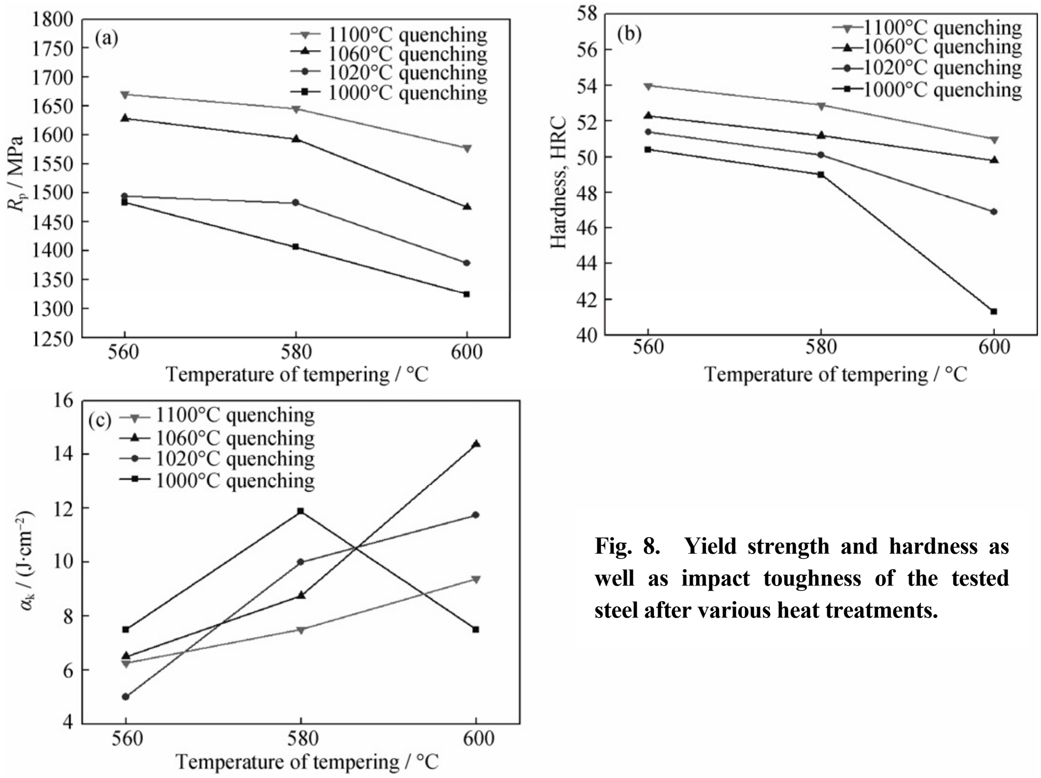

Fig. 8. Yield strength and hardness as well as impact toughness of the tested steel after various heat treatments.

As shown in Table 4, compared with the tensile properties of nitrogen-free H13 die steel [16], the tensile strength and yield strength of the nitrogen-containing die steel are much higher, which are greatly improved by 25.7% and 16.1%, respectively. Their elongation and reduction of area are approximate.

Table 4. Comparison of room-temperature tensile properties between H13 steel and the tested steel

Steel	R_m / MPa	R_p / MPa	A / %	Z / %
Nitrogen-free H13 die steel	1496.00	1310.00	6.00	24.90
Tested steel	1880.79	1521.32	8.40	24.84

4. Conclusions

(1) Nitrogen expands the γ-phase region and may decrease the phase transformation temperatures of die steels. The Ac_3, Ac_1, Ms, and Mf temperatures of the nitrogen-containing die steel are 847, 800, 307, and 180°C, respectively, which are lower than that of AISI H13 steel.

(2) By means of isothermal spheroidal annealing, the nitrogen-containing die steel is austenitized at 850-930°C, followed by spheroidizing at 700-750°C and cooling to room temperature. The homogeneous and stable globular pearlite microstructure and proper hardness with good machinability can be obtained.

(3) The appropriate quenching temperature of the nitrogen-containing die steel in the paper is 1020-1060°C. The contents of carbon, nitrogen, and other alloying elements in quenched martensite increase due to their supersaturated solid solution into the matrix, which can improve the

strength and hardness of the nitrogen-containing steel.

(4) After the tested steel quenched at the same temperature, increasing the tempering temperature can improve the ductility and toughness; however, the strength and hardness may decrease deeply. When the nitrogen-containing steel is tempered between 560 and 600°C, the precipitations of fine alloy carbides result in good tempering stability, high strength, and a certain ductility and toughness.

(5) Nitrogen element can elevate the strength and hardness of die steels substantially, while the ductility and toughness may hold in a high level, such as the impact toughness is over 10 J·cm^{-2}.

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