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ARTICLE

Effect of Solution Treatment on Microstructures and Mechanical Properties of High Nitrogen Stainless Steel

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Abstract: High nitrogen stainless steel has excellent mechanical and chemical properties. Cr-Mn-Mo high nitrogen stainless steel with a nitrogen content of 0.54wt% was smelted by increasing the content of Cr and Mn at the nitrogen partial pressure of 80 000 Pa. The sample steels after hot rolling were held at 800, 900, 1000, 1100 and 1200 °C for 1, 2, 3, 4, and 5 h. Orthogonal analysis was carried out to study the microstructure, yield strength, tensile strength, elongation to fracture, reduction of area, and product of strength and plasticity under different temperatures and holding time, in order to find the best heat treatment temperature and time for the test steel. The results show that Cr_2N precipitates in the samples without solution treatment and after solution treatment at 800 and 900 °C, and ferrite precipitates in the samples after solution treatment at 1200 °C. The materials treated at 1000 and 1100 °C are pure austenite. The specimen held at 1000 °C for 4 h has the best plasticity and high strength, and its section shrinkage and post-fracture elongation can reach 67.5% and 69.5%, respectively. The strength of the samples without heat treatment is the highest, and the reduction of section and elongation after fracture remains at 42% and 49.9%. The comprehensive mechanical properties of the samples held at 1000 °C for 1 h are the best, and the product of strength and plasticity can reach 58.59 GPa%.

Key words: high nitrogen stainless steel; solution treatment temperature; solution treatment time; strength; plasticity

With the development of science and technology, stainless steel is required to have the characteristics of high-cost performance, green and environmental safety, and the development of low-cost stainless steel with good comprehensive performance is of great significance to the development of the steel industry. Because Ni is scarce and expensive and is harmful to the human body, researchers have replaced the rare and expensive Ni elements with inexpensive N and Mn elements, and Cr-Mn series high-nitrogen stainless steels have been successively developed[1,2]. In order to further improve the corrosion resistance of steel, the Cr-Mn-Mo series high nitrogen stainless steel was developed. Nitrogen exists in steel in the form of interstitial solid solution, which can greatly increase the strength of steel and has good plasticity and toughness[3-5]. Because high-nitrogen stainless steel has the characteristics of high strength, excellent plasticity, and good pitting resistance, it has the potential to replace traditional nickel-based stainless steel and be used in marine and military equipment. A large number of studies have shown[6-9] that the optimization of material structure and mechanical properties can be achieved through heat treatment. Although many scholars have studied high-nitrogen steel[10-14], the research on the effect of different solid solution temperatures and time of high-nitrogen stainless steel on its comprehensive mechanical properties is insufficient. This study investigated the structure of the sample steel after solution treatment at 800, 900, 1000, 1100, 1200 °C for 1, 2, 3, 4 and 5 h, as well as yield strength, tensile strength, elongation to fracture, reduction of area, and product of strength and plasticity to determine the effect of different treatment processes on the comprehensive mechanical properties of the test steel and to find the best solution

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treatment temperature and time.

1 Experiment

The tested steel was smelted with industrial pure iron, electrolytic manganese, pure chromium, ferromolybdenum, nitrogen-manganese alloy, and nitrogen-chromium alloy in a 50 kg vacuum induction furnace at a nitrogen partial pressure of 80 000 Pa and a temperature of 1550 °C, and a small amount of aluminum was added for de-oxidation. After tapping, it is cooled to room temperature with the furnace to form a 150 mm×150 mm square billet. After electroslag remelting, the chemical composition of the material is shown in Table 1, and then it is hot-rolled into a hot-rolled sheet with a width of 100 mm and a thickness of 10 mm.

The 10 mm×10 mm metallographic specimens and standard tensile specimens of the hot-rolled sheet are shown in Fig. 1. The specimens were kept at room temperature, 800, 900, 1000, 1100 and 1200 °C for 1, 2, 3, 4, and 5 h, and then water cooled to room temperature. The metallographic specimens were polished by 60#, 400#, 800#, 1200#, 1500#, and 2000# sandpapr and then polished with polishing agent (granularity 0.5) to a mirror surface without scratches, and then subjected to XRD inspection (the diffraction angle is 10°~100°). The standard XRD patterns were compared, and the composition was determined. Afterward, the grain boundaries were corroded by aqua regia (concentrated hydrochloric acid: concentrated sulfuric acid=3:1), and the morphology and size of the structure were observed. The tensile specimens were polished with sandpaper from 60# to 800#, and the CMT4204 static hydraulic universal testing machine was used for tensile testing to determine the tensile strength, yield strength, reduction of area, elongation to fracture, and product of strength and plasticity. To determine the best solution treatment temperature and time for the test steel, the implementation standard GB/T228.1-2010 was used.

2 Results and Discussion

2.1 Effect of solution temperature on microstructure

FactSage software was used to calculate the phase diagram of the alloy, as shown in Fig.2. Fig.2 shows that the alloy is a single austenite phase at 920~1124 °C; when the temperature is higher than 1124 °C, the ferrite phase is gradually precipitated from the austenite; when the temperature is lower than 920 °C, the close-packed hexagonal crystals phase is gradually precipitated from the austenite. After XRD analysis, it can be determined that the precipitated close-packed hexagonal crystals phase is Cr_2N.

Table 1 Chemical composition of the Fe-Cr-Mn-Mo-N steel (wt%)

Cr	Mn	Mo	C	N	Fe
17.88	15.24	1.41	0.009	0.54	Bal.

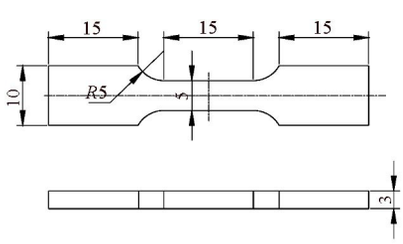

Fig.1 Tensile spcimen structure drawing

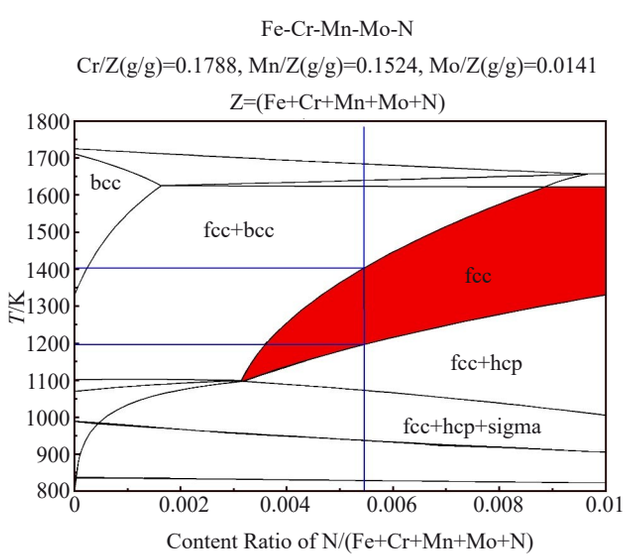

Fig.2 Phase diagram of test steel

Fig.3 and Fig.4 show the XRD patterns and microstructures of the specimens kept at room temperature, 800, 900, 1000, 1100, and 1200 °C for 1, 3, and 5 h. It can be seen from them that the structure of the test steel without solution treatment is fine austenite but there are a small number of precipitates. According to the XRD pattern, it can be determined that the precipitate is Cr_2N, and the crystal grains are very small, resulting in extremely large grain boundaries. There are many grains with high-density and different orientation; after holding at 800 °C for 1 h, the grains increase from 5 μm to 9 μm, but the grains are still small, and the precipitates in the grain boundaries increase significantly; after holding at 900 °C, the grain size increases, about 14 μm, the grain boundary density decreases, and there is still a small amount of Cr_2N precipitates; after the heat preservation at 1000 °C, the crystal grains increase significantly to about 30 μm, the twins are obviously reduced, and the precipitates Cr_2N are almost all dissolved, which is a pure austenite structure; after 1100 °C heat preservation, the crystal grain further increases to about 50 μm, the twins are reduced, and the austenite structure is still maintained. After holding at 1200 °C, the grain size increases again, about 65 μm, and the ferrite structure begins to precipitate at the grain boundary. Fig. 5 shows the average grain size after holding for 1 h at different temperatures.

In summary, with the increase of the holding temperature, the crystal grains gradually become larger, the grain boundary density and twin crystals gradually decrease. This is because high temperature promotes the growth and reorganization of

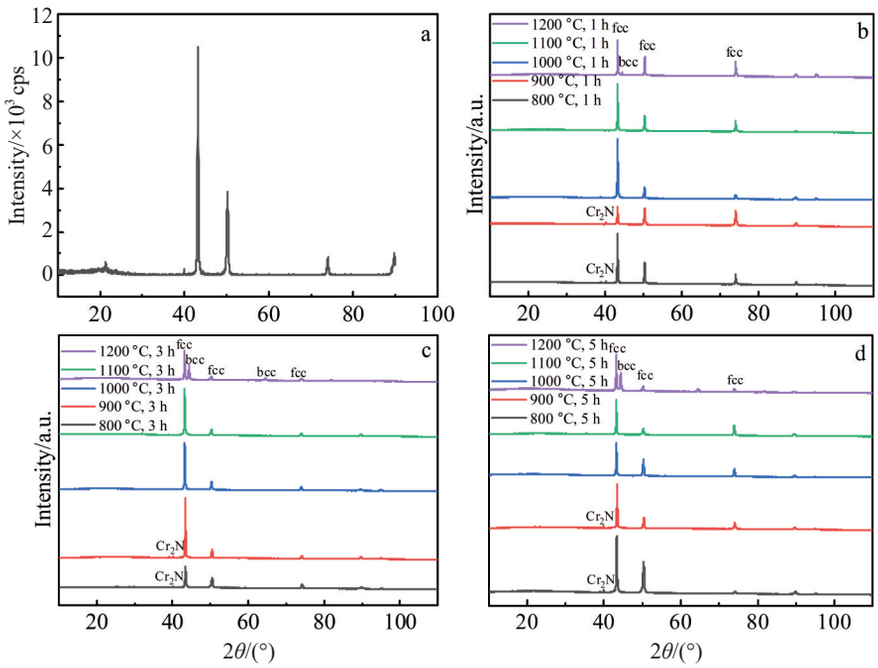

Fig.3 XRD patterns of test steels held at room temperature (a) and 800, 900, 1000, 1100 and 1200 °C for 1 (b), 3 (c), and 5 h (d)

the crystal grains. At room temperature, 800 and 900 °C, it is an austenite structure with a small amount of Cr_2N precipitation. The precipitation of Cr_2N inhibits the growth of grains to a certain extent, so the grains grow slowly below 900 ° C; at 1000 and 1100 ° C, Cr_2N dissolves into the austenite, and the grains grow quickly which are fully austenitic structures. When the temperature reaches 1200 ° C, ferrite will begin to precipitate from the austenite, which is basically consistent with the phase diagram in Fig.2.

2.2 Effect of solution time on microstructure

Fig. 4 and Fig. 6 show the influence of the microstructure and the holding time on the grain size at 800, 900, 1000, 1100, and 1200 ° C for 1, 3, and 5 h. It can be seen that as the holding time increases, the crystal grains show a tendency to become larger, the growth rate gradually decreases, and there are fewer and fewer twins; the precipitates at 800, 900, and 1200 ° C increase with the holding time, and the growth is gradually uniform, because the high temperature promotes the growth and reorganization of the crystal grains. In addition, with the extension of the holding time at 800 ° C, more and more Cr_2N precipitate between the grain boundaries; due to the influence of phase balance, the Cr_2N between the grain boundaries at 900 °C after 3 h is significantly less than that at 800 ° C; Cr_2N is almost completely dissolved at 1000 and 1100 ° C, and it is pure austenite; at 1200 ° C, ferrite is gradually precipitated from austenite, and with the extension of holding time, the precipitated ferrite gradually increases.

2.3 Effect of solution temperature on mechanical properties

Tensile tests were carried out on test steels with different solution treatment temperatures at room temperature to reveal the influence of different solution treatment temperatures on the mechanical properties of the test steels. To show the effect of heat treatment temperature on the strength and plasticity of the material more intuitively, the composite alloy was heated to 800, 900, 1000, and 1100, 1200 °C for 1, 2, 3, 4, and 5 h, and then water cooled to room temperature. After the stretching data, the Origin software was used to obtain to Fig.7 and Fig.8.

Fig.7 shows the sample's tensile strength and yield strength after heat preservation and water cooling at different temperatures. It can be seen that the tensile strength and yield strength of the sample without solution treatment can reach 1157 and 884 MPa, respectively. When the holding temperature is between room temperature and 800 ° C, the broken line shows a slow downward trend as the holding temperature increases. When the holding temperature is 800~1000 °C, the broken line shows a rapid downward trend as the holding temperature rises. When the holding temperature is 1000~1100 °C, the strength decreases with the increase of the holding temperature. When the holding temperature is 1100~1200 ° C and the holding time is 1~2 h, the strength will decrease with the increase of the holding temperature, and the strength will increase when the holding time is 3~5 h. And the intensity drops fastest in the 800~1000 °C range.

In the process of plastic deformation of steel materials, the main way is the slip of dislocations. The grain boundary has an obstructive effect on the slip of dislocations. The fine grains have a large number of dislocations, which hinder dislocation movement. It has a greater effect and can significantly increase the strength of steel; fine precipitates (ferrite, Cr_2N) increase the distortion of the crystal lattice, have a pinning effect on the slip of dislocations, and can

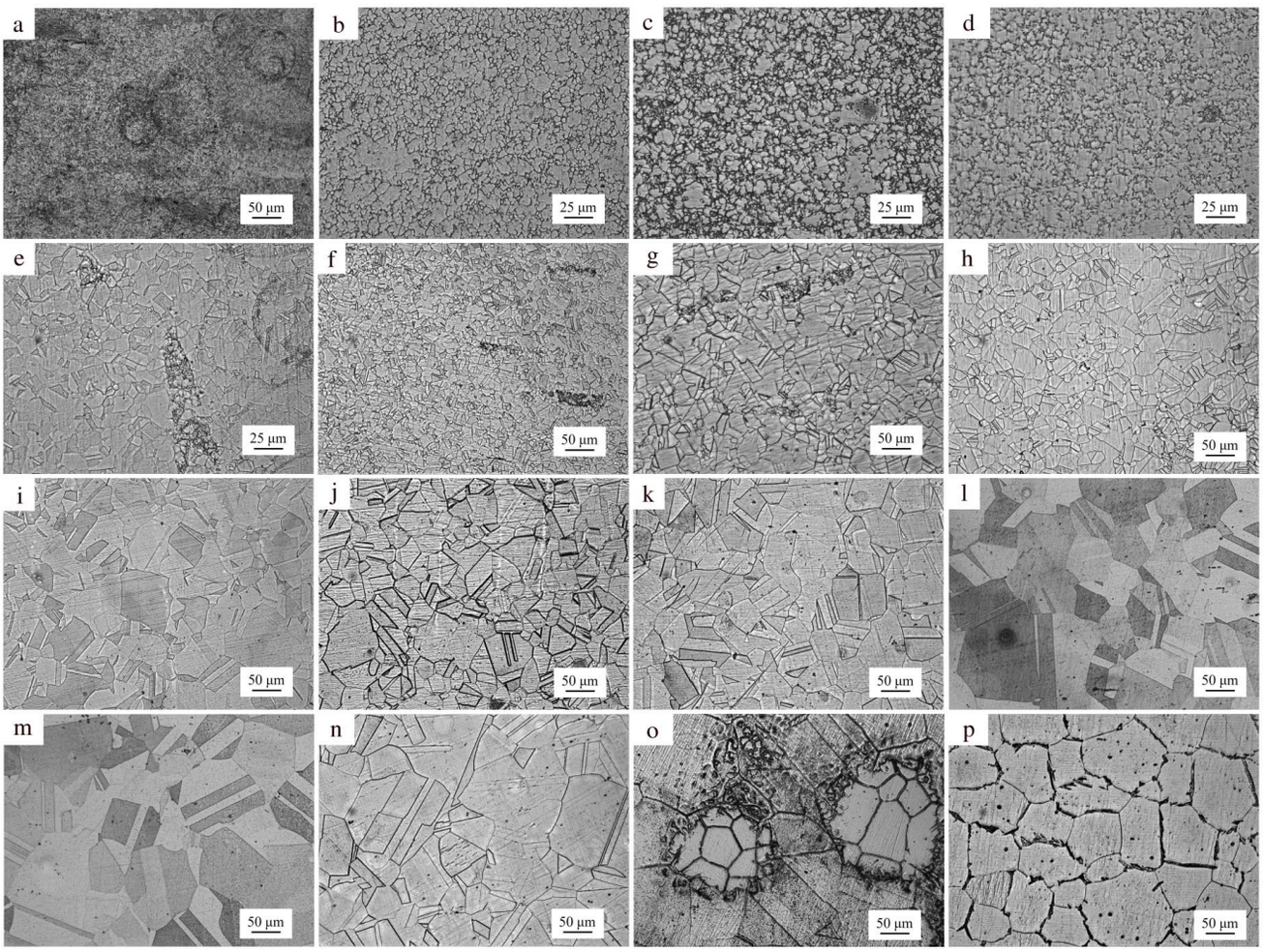

Fig.4 Microscopic structures of test steel solution treated under different conditions: (a) untreated; (b, c, d) 800 ℃; (e, f, g) 900 ℃; (h, i, j) 1000 ℃; (k, l, m) 1100 ℃; (n, o, p) 1200 ℃; (b, e, h, k, n) 1 h; (c, f, i, l, o) 3 h; (d, g, j, m, p) 5 h

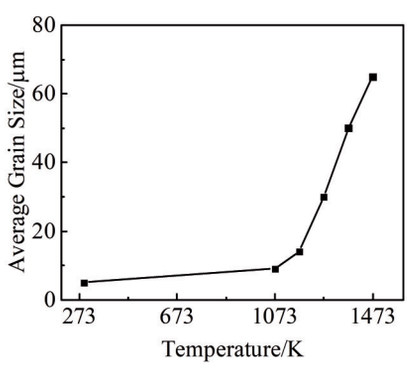

Fig.5 Grain size of test steels after holding at different temperatures for 1 h

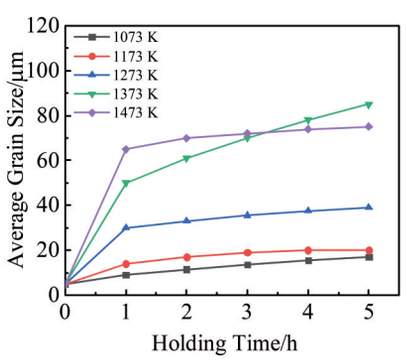

Fig.6 Effect of holding time on average grain size of test steels

increase the strength of steel.

At room temperature, the crystal grains are extremely small, the grain boundary density is extremely high, and the strength is the highest; although a small amount of Cr_2N precipitates at 800 °C which play a role of strengthening the second phase, the increase in grain size and the decrease in grain boundary density still dominate, making the strength slightly decrease; the strength decreases significantly at 800~1000 °C, which is due to the gradual dissolution of Cr_2N as the holding temperature rises to the austenite phase region, so that the grain boundary density and the second phase precipitates are significantly reduced. At 1000~1100 °C, the growth of grains leads to a decrease in grain boundary density and reorganization of grains leads to a decrease in dislocations and twins, resulting in a slow decline in strength. At 1100~

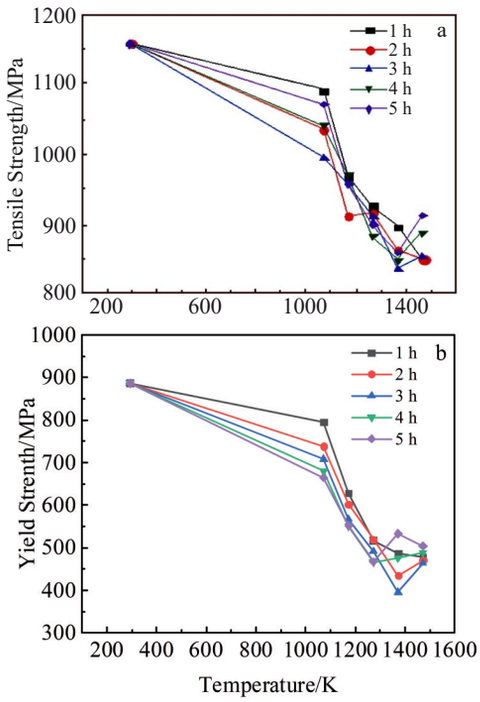

Fig.7 Tensile strength (a) and yield strength (b) of test steels after solution treatment at different temperatures

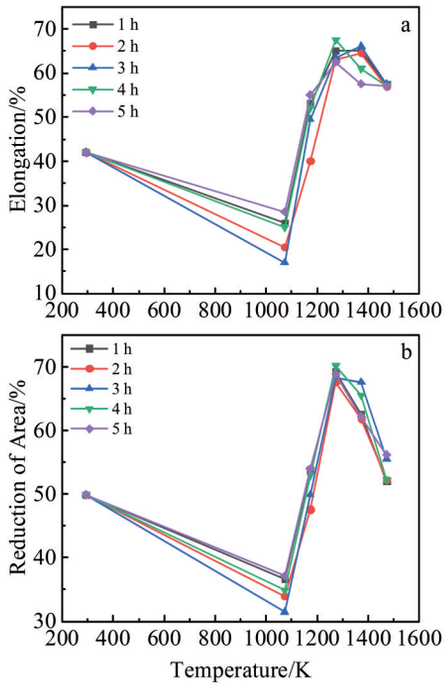

Fig.8 Elongation (a) and reduction of area (b) of test steels after solution treatment at different temperatures

1200 °C, on the one hand, the grains grow, which reduces the strength, on the other hand, ferrite gradually precipitates from the austenite, and the second phase strengthening effect appears again, making the strength increase. The combined effect of two factors makes the intensity basically stable or even rise. When the holding time reaches more than 3 h, the austenite grains grow to the critical value and basically stop growing. At this time, the precipitation and growth of austenite ferrite play a pinning role to a certain extent, so the strength increases.

Fig. 8 shows the elongation and reduction of the area of the experimental steel after heat preservation and water cooling at different temperatures. It can be seen that the plasticity is the best at 1000~1100 °C, and the elongation and reduction of the area can reach 67.5% and 69.5%, respectively. At room temperature~800 °C, both the elongation and shrinkage show a downward trend with the increase of the holding temperature, mainly because Cr_2N gradually precipitates from the austenite during the holding at 800 °C, which reduces the plasticity of the steel. At 800~1000 ° C, as the holding temperature gradually approaches the temperature of the single austenite phase zone, the Cr_2N content gradually decreases, and the elongation and cross-sectional ratio increase significantly. At 1100~1200 °C, the austenite grains gradually precipitate Fe, With the increase of the element body, the plasticity of the test steel shows a downward trend.

2.4 Effect of solution time on microstructure

Fig. 9 shows the tensile strength and yield strength of the test steel under different holding conditions. It can be seen

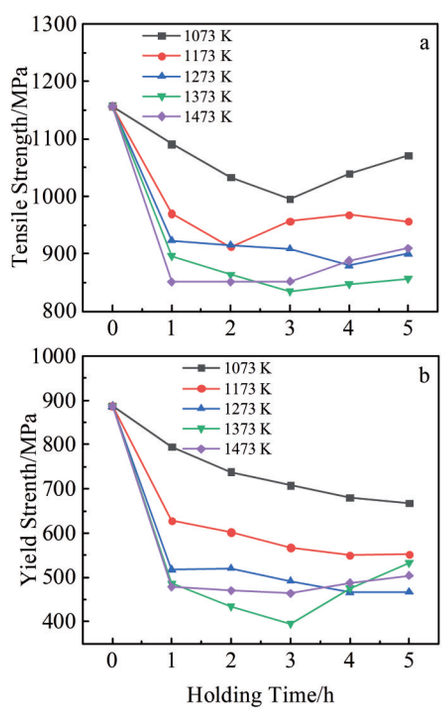

Fig.9 Tensile strength (a) and yield strength (b) of test steels after solution treatment at different temperatures for different time

that at any temperature, with the extension of the holding time, the strength tends to drop sharply, then gradually stabilizes and rises. This is due to the following reasons with

the extension of the holding time: (1) the grain boundary density and the strength decrease due to the growth of crystal grains; (2) the crystal lattice reorganization reduces the number of defects and twins, and the strength decreases; (3) the precipitates cause a solid solution strengthening effect and the strength decreases; (4) the precipitates increase, and the strengthening effect of the second phase has a pinning effect which can increase the strength; (5) the precipitation of the grain boundary is gradually evenly distributed[15-17] and the second phase strengthening effect occurs to increase the intensity. In the early stage of heat preservation, the crystal lattice growth of reason (1) and (2) are dominant, making the strength drop sharply; as the heat preservation time increases, the grain size gradually tends to balance, and the driving force for grain growth becomes smaller and gradually loses the dominant state. The status decrease the strength slowly until stable; as the holding time is extended again, the grain size gradually tends to balance, and reason (4) and (5) take the leading role[18-22], making the strength of the test steel show a slight upward trend. In addition, the higher the temperature, the better the kinetic conditions of the grain transformation. Compared with the low temperature (800, 900 °C), the high temperature (1100, 1200°C) samples can enter into the middle and late stages earlier.

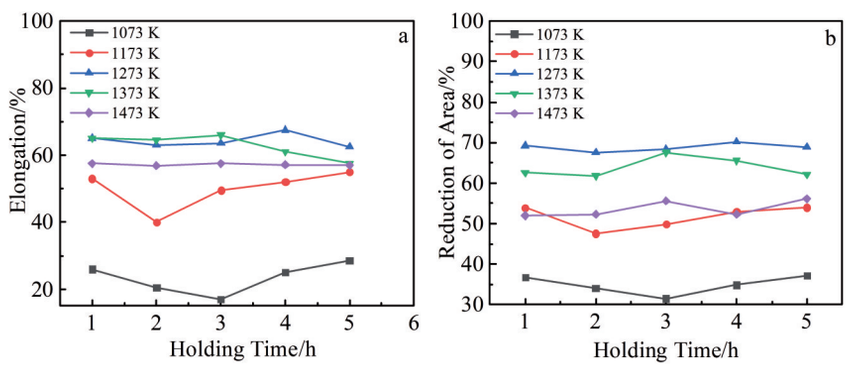

Fig.10 Elongation (a) and reduction of area (b) of test steels after solution treatment at different temperatures for different time

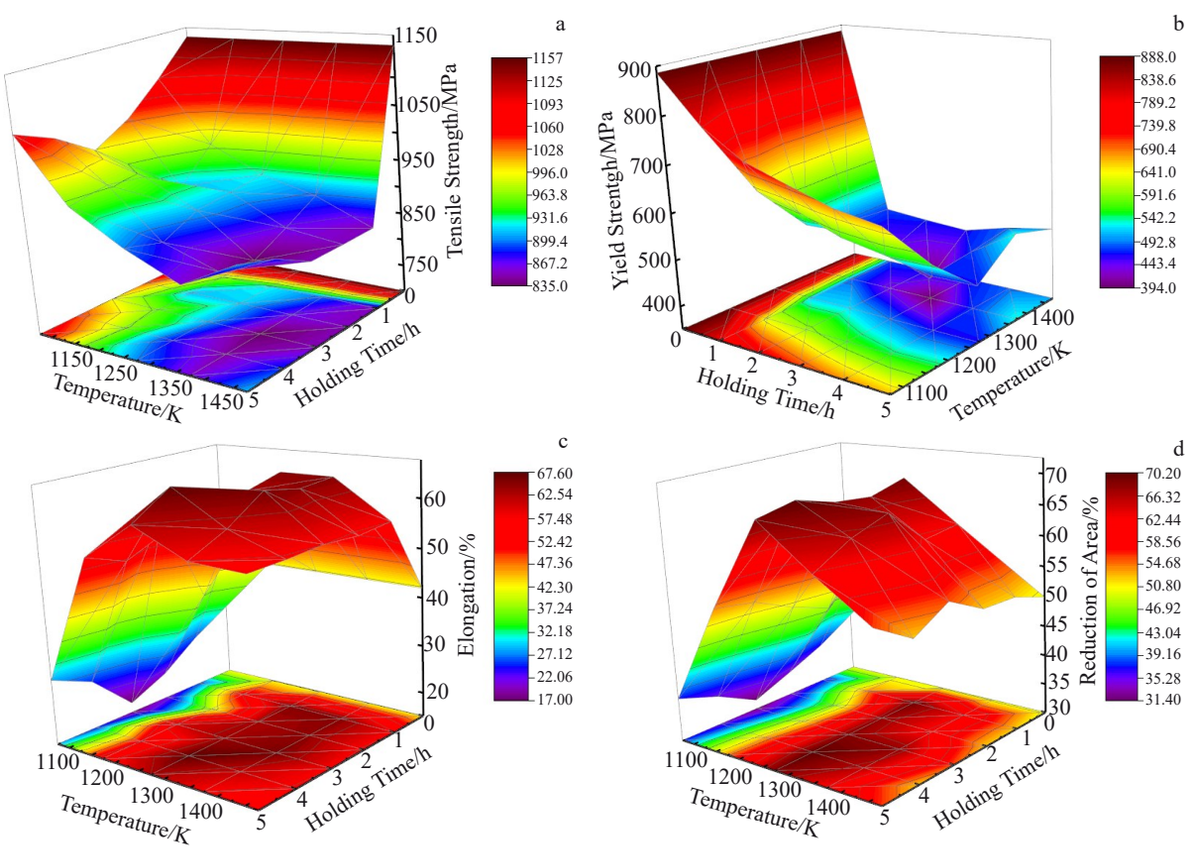

Fig.11 Combined effect of solution time and temperature on tensile strength (a), yield strength (b), elongation (c) and reduction of area (d)

Fig. 10 shows the elongation and reduction of area of the test steel under different holding conditions. It can be seen that during solution treatment at 800 and 900 ° C, the elongation and shrinkage of the area both decrease first and then increase with the increase of holding time. This is because the precipitation of Cr_2N at the initial stage of the holding causes the plasticity to decrease, and then the precipitation reaction gradually tends to an equilibrium state. With the extension of holding time, the grains and precipitates are gradually distributed uniformly, which improves plasticity[23,24]. There is no phase change when held at 1000 and 1100 °C, the grain size is close to equilibrium after 1 h and the length is very small, so there is almost no change in plasticity with the extension of holding time. At 1200 ° C, ferrite precipitates, but due to the higher temperature, the precipitated ferrite is fine and uniform, resulting in little plastic change, which has a small effect on plasticity, and the elongation and reduction of the area remain basically stable.

The effect of solution time and temperature is displayed in Fig. 11. The strength of the sample without solution treatment is the highest, its yield strength and tensile strength can reach 1157 and 884 MPa, respectively, and its elongation and reduction of area are still maintained at 42% and 49.9%, respectively. The sample has the best plasticity at 1000 °C for 4 h, the elongation and the reduction of area can reach 67.5% and 69.5%, respectively, and the tensile strength and yield strength can still be maintained at 880 and 466 MPa, respectively. Considering comprehensively, the comprehensive mechanical properties are the best after heat preservation at 1000 °C for 1 h. Its elongation and reduction of area are 65% and 69.17%, respectively, and the yield strength and tensile strength are 923 and 517 MPa, respectively. Its product of strength and plasticity can reach 58.59 GPa%.

3 Conclusions

1) With the increase of the solid solution temperature, the crystal grains gradually increase, and Cr_2N precipitates at 800~900 °C, which is single austenite structure at 1000~1100 °C, and accompanied by ferrite at 1200 °C.

2) With the extension of the solution time, the crystal grains grow up and gradually stabilize, and the precipitates gradually increase and tend to balance.

3) As the solid solution temperature rises, the strength of the test steel gradually decreases, and the plasticity shows a trend of first rising and then falling, and the plasticity is the best at the austenite temperature.

4) With the extension of the solution time, the strength of the test steel shows a sharp decline first to a steady state. The plasticity at 800 and 900 °C declines first and then rises, and the extension of the holding time at 1000, 1100 and 1200 °C has an small effect on the plasticity.

5) The maximum yield strength and tensile strength of the test steel without solution treatment can reach 1157 and 884 MPa, respectively, and the plasticity of the sample after holding at 1000 ° C for 4 h is the best, its elongation and reduction of the area can reach 67.5% and 69.5%, respectively. Sample treated at 1000 °C for 1 h has the optimal comprehensive mechanical properties. Its product of strength and plasticity can reach 58.59 GPa%.

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固溶处理对高氮不锈钢微观组织及力学性能的影响

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摘　要：高氮不锈钢具有优异的综合性能。通过增加铬、锰含量在氮分压为80 000 Pa下成功冶炼出氮质量分数为0.54%的Cr-Mn-Mo系高氮不锈钢；试样钢热轧后分别经800、900、1000、1100、1200 ℃保温1、2、3、4、5 h固溶处理后正交分析，研究在不同温度和保温时间下的组织、屈服强度、抗拉强度、断后延伸率、断面收缩率和强塑积，旨在找到试验钢最佳的热处理温度和时间。结果表明，未经固溶处理和经800、900 ℃固溶处理后的试样中有Cr_2N析出，1200 ℃固溶处理后试样中析出铁素体，1000、1100 ℃固溶处理的试样为纯奥氏体组织，且在1000 ℃下保温4 h的试样塑性最好并有较高的强度，其断面收缩率和断后延伸率分别可以达到67.5%和69.5%。未经热处理的试样强度最高，并且断面收缩率和断后延伸率仍然保持在42%和49.9%。在1000 ℃下保温1 h的试样综合力学性能最好，强塑积可达到58.59 GPa%。

关键词：高氮不锈钢；固溶处理温度；固溶处理时间；强度；塑性

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