

Article

Precipitation Behavior of the Second Phase in High-Nitrogen Austenitic Stainless Steel Alloyed with V and Nb

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Abstract: The retaining ring is one of the components with the highest stress on the rotor of the turbine generator. Due to the high content of alloying elements in this type of steel, it is extremely easy to precipitate a large amount of the second phase during the forming process of a material, which deteriorates the processability. Therefore, it is very important to study the second phase precipitation of high-nitrogen steel. This work addresses the precipitation behavior of the second phase in a high-nitrogen austenitic stainless steel alloyed with V and Nb. The optical microscopy, scanning electron microscopy, transmission electron microscopy and X-ray diffractometry were applied to investigate the influence of heat treatment parameters on the morphology and composition of the precipitated particles. The addition of V and Nb results in the formation of NbVN and VN particles, of which the amount is slightly decreased after a subsequent solution treatment at 1200 °C for 1 h. The results demonstrate that an additional aging treatment at 800–1000 °C for three leads to the participation of lamellar Cr₂N and the undissolved MX phase. The content of the precipitates increases with increasing both the aging temperature and the aging time. The addition of V/Nb elements effectively improves the stability of MX phase, and the existence of high energy zones (the MX phase interface and grain boundary) promotes the early formation of Cr₂N. Cr₂N is shown to nucleate in the high energy zones, which thereafter grows into austenite grains, demonstrating a normal discontinuous cellular growth.

Keywords: high-nitrogen austenitic steel; nitrogen; precipitation; microstructure



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1. Introduction

High-Nitrogen Austenitic Stainless Steel (HNASS) is a new type of metal material which is nickel-saving and bio-friendly with excellent mechanical properties and corrosion resistance [1]. Owing to the above advantages, in recent years, it has been widely used in various fields such as machinery, chemical industry, energy and electricity, biomedicine and jewelry [2–4] and is also a potential candidate in the manufacturing of armor [5]. P900 (18Cr-18Mn-0.6N), P900-N (18Cr-18Mn-0.9N) [6] and P2000 (16Cr-14Mn-3Mo-0.9N) [7] steels are the typical industrially used high-nitrogen austenitic stainless steel employed in the manufacturing of large generator guard rings [8]. Due to the high content of alloying elements in this type of steel, it is extremely easy to precipitate a large amount of the second phase during the forming process of a material (high-temperature pressure processing, welding, etc.) [9], which deteriorates the processability [10].

In recent years, extensive research has been directed towards the second phase precipitation behavior of HNASS [1], especially concerning Mn18Cr18N steel with the obtainment of excellent results. Regarding intermetallic phases (σ , η , etc.), Lee [11] and Ma et al. [12] proposed that the σ phase can be transformed from γ or χ phase, accompanied by the

diffusion and transfer of N element. Moreover, increasing the content of Mo element in HNASS promotes the precipitation of χ and η phases [13,14]. In addition, research activities focusing on the precipitation of carbonitrides such as M_2X , $M_{23}C_6$ and M_7C_3 are also implemented [15]. Hu et al. [16] pointed out that with an increase in the nitrogen content in HNASS, the precipitation of $M_{23}C_6$ during the hot stretching process can be promoted, and $M_{23}C_6$ appears at the grain boundaries, leading to intergranular cracking easily [17]. Apparently, $M_{23}C_6$ has a detrimental effect on the hot forming of HNASS.

As one of the most popular second phases in HNASS, Cr_2N is a brittle phase with a closely packed hexagonal structure. As stated in [18,19], irrespective of the initial processing state of the Mn18Cr18N steel, Cr_2N as well as a small amount of $Cr_{23}C_6$ develops during solution and aging treatments. With decrease in the C/N mass ratio, the precipitation of Cr_2N becomes more sensitive, while the precipitation of $Cr_{23}C_6$ is gradually retarded [20]. Generally, Cr_2N is prone to precipitate at the grain boundaries and grows into crystal grains in a discontinuous cellular growth type such that forming the “nitrogen-containing pearlite” with unbalanced austenite [21]. The presence of a large amount of Cr_2N seriously deteriorates the plasticity of HNASS, which is not conducive to plastic pressure forming [22,23]. Kartik et al. [24] demonstrated that during high-temperature aging, the precipitation of Cr_2N and other intermetallic phases is accompanied by the consumption of N, Cr, Mn and other elements in the solid solution. As a result, the strength and hardness of HNASS decrease.

Previous research shows that the addition of V and Nb in HNASS has measurable influence on the second phase precipitation behavior of HNASS, which thereafter affects performance and serviceability (mechanical properties, stress corrosion resistance, etc.). Knutsen [25] et al. believed that the addition of V and Nb can regulate the association between M_2X and MX phases during precipitation. Among them, V improves the stability of M_2X phase, and Nb inhibits the stability and kinetics of M_2X phase. For the MX phase, Knutsen believes that Nb promotes the formation of MX -type precipitation during solidification and aging, while V participates in the reaction of MX -type precipitation formation, but its role is not clear. Therefore, in this study, the precipitation behavior of the second phase in HNASS was studied by the solution aging method. The second phase precipitation process in HNASS is studied [22].

2. Materials and Experimental Methods

The experimental HNASS was smelted using a Dming Technology 40 kg induction electric furnace and cast into steel ingots at 1500~1700 °C. To improve the compositional uniformity of the material, the ingot was electro slag remelted. Finally, the chemical constituents of HNASS were obtained as shown in Table 1. The HNASS after electros slag remelting was forged into a round rod-shaped sample with a diameter of 30 mm, referred to as the original sample.

Table 1. Chemical composition of test materials (wt. %).

Element	C	Si	Mn	Cr	Mo	Nb	V	N	S	P	Fe
Content (wt.%)	0.06	0.63	15.66	18.07	3.02	0.07	0.66	1.02	0.005	0.009	margin

The rectangular parallelepiped samples with size of 5 mm × 5 mm × 10 mm were cut at the center of the round bar and solution heat treated at 1200 °C for 1 h with following water cooling. Aging of the solid solution treated samples was achieved by isothermal heating at 600, 800, 900 and 1000 °C for 0.5~5 h and the subsequent water-cooling. The original forged state samples, solid solution samples and aging samples were pre-grinded, polished and etched using a mixture of 2.5 g $CuCl_2$ + 100 mL HCl + 100 mL C_2H_5OH (solution). The morphology was observed by a Leica DM6M optical microscope (OM), FEI Scios03040702 focused ion beam field emission scanning electron microscope (SEM) and FEI Tecnai G2F20S-TWIN transmission electron microscope (TEM). The elemental

composition of the precipitates was characterized by energy dispersive spectroscopy (EDS) with an SEM and TEM. A D/MAX2500PC03030502 X-ray diffractometer (XRD) was used to analyze the phases of the original and solid solution samples.

3. Results

3.1. The Second Phase of the Original HNASS before Heat Treatment

The microstructure of the original forged HNASS in Figure 1a displays that the metallurgical structure of the original forged HNASS is composed of austenitic grains of different sizes and granular second-phase particles dispersed onto the grains. The XRD analysis of the original structure (Figure 1b) demonstrates that the as-produced HNASS sample is composed of mainly austenite and nitrides (i.e., NbVN or VN). The SEM photograph in Figure 1c clearly shows that secondary particles in a special shape are irregularly preferentially distributed in the austenite grains rather than boundaries, consistent with the metallographic analysis in Figure 1a. The EDS surface scanning of the granular second phase exhibits that the elemental composition of the particles is poor in Fe and Mn and rich in N and V as compared to in the austenite matrix. Accordingly, these particles are VN precipitates rather than σ -equal intermetallic consisting of Fe and Mn.

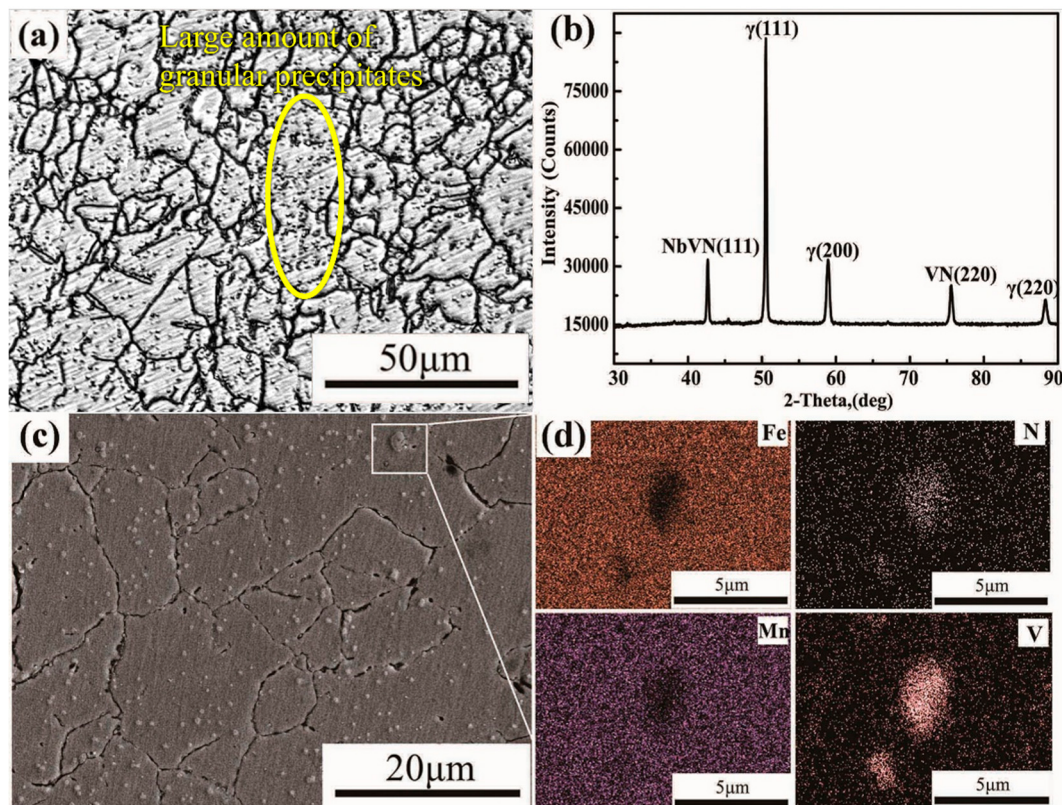


Figure 1. Metallographic structure of the original sample (a): dispersed granular second phase; XRD pattern (b): indicates that the precipitates are NbVN and VN; SEM image (c); EDS surface scan of the elemental distribution of the second phase-VN (d).

3.2. The Second Phase in Solution-Treated HNASS

Figure 2a shows the X-ray diffractogram of the solution heat-treated sample. Similar to that of the as-produced sample, the phase structure is characterized by the predominant austenite and a small amount of V and Nb alloyed nitrides. The intensity of the diffraction peaks of precipitates, however, is lower compared to the as-produced sample, indicating that VN particles are not completely dissolved after solution heat treatment. The undissolved second phase is observed in the TEM image of the solution-treated HNASS

(Figure 2b), which confirms the existence of the second phase precipitates in a solution heat treated V and Nb alloyed HNASS.

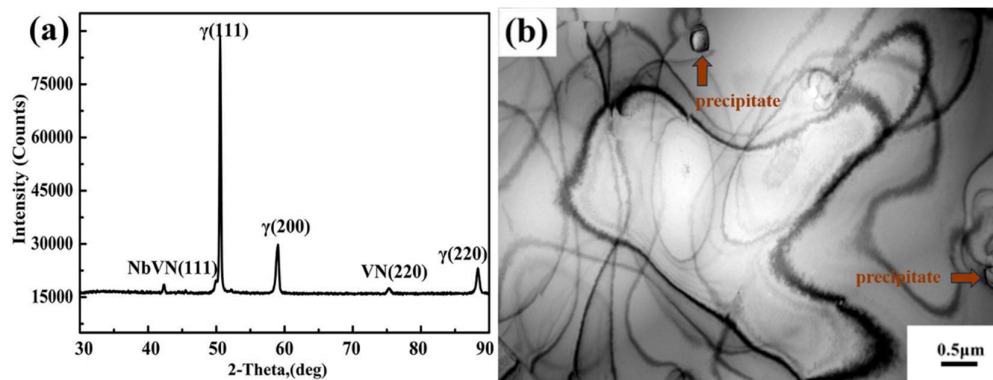


Figure 2. XRD analysis of the HNASS solid solution (a): the content of precipitates decreases and TEM image (b): small amounts of undissolved precipitates were found.

The TEM image of the solid solution HNASS in Figure 2b confirms the co-existence of two types of second phases showing different dimensions. As shown in Figure 3a–c, the dimension of the relatively large precipitate is about 200 nm, while the other small precipitates with a size of about 50 nm are present in spherical shape. The massive second phase in Figure 3a was subjected to SAED calibration (Figure 3b), and the MX phase was determined with a simple cubic structure and a lattice constant of $a = b = c = 0.413$ nm with the actual chemical formula, VN. A comparison of the EDS results between the two precipitates (Figure 3d) displays that the types of elements contained in the two second-phase precipitates are the same, but the content of each element is slightly different. Despite a significant difference in size, the two precipitates as observed are both in the MX type.

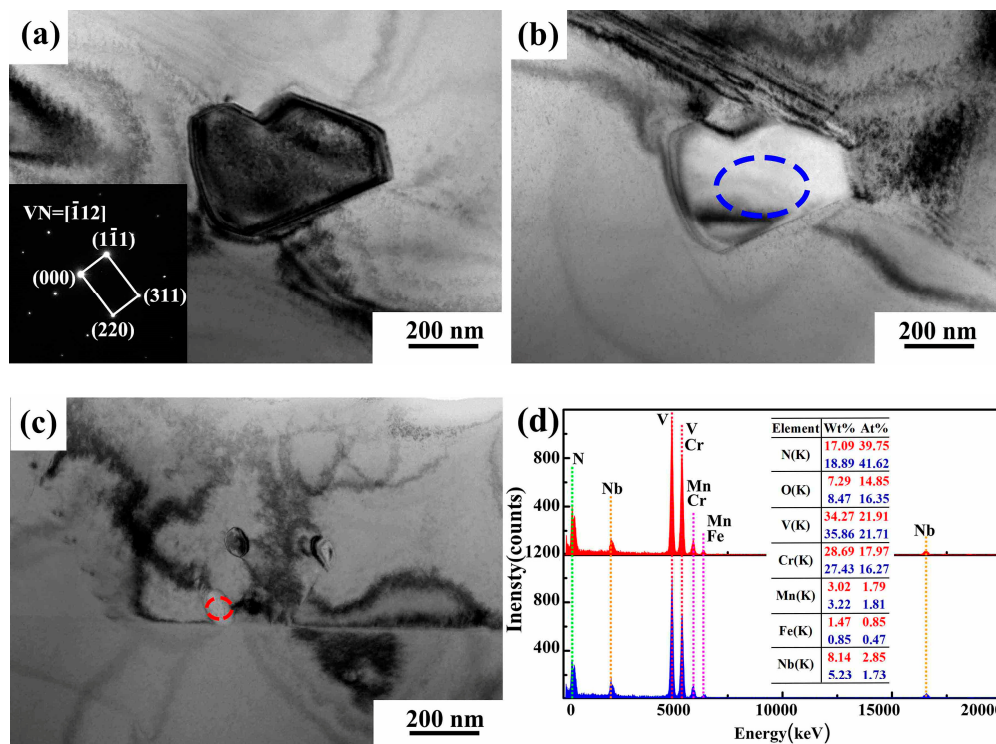


Figure 3. TEM image and EDS analysis of the second phase in solid solution HNASS after 1 h at 1200 °C: the addition of Nb and V elements results in the precipitation of MX-type nitrides.

3.3. Precipitated Phases in Aging HNASS

3.3.1. Precipitated Phase after 3 h of Aging

Aging treatment at different temperatures leads to remarkable differences in the morphologies of HNASS. The SEM micromorphology of HNASS after isothermal aging at 600–1000 °C for 3 h is shown in Figure 4, where the morphology of each precipitate is marked. It is noted that with increasing aging temperature, the content of the precipitated phase in HNASS gradually increases, and in particular, clustered lamellar based on the dispersed particles is present.

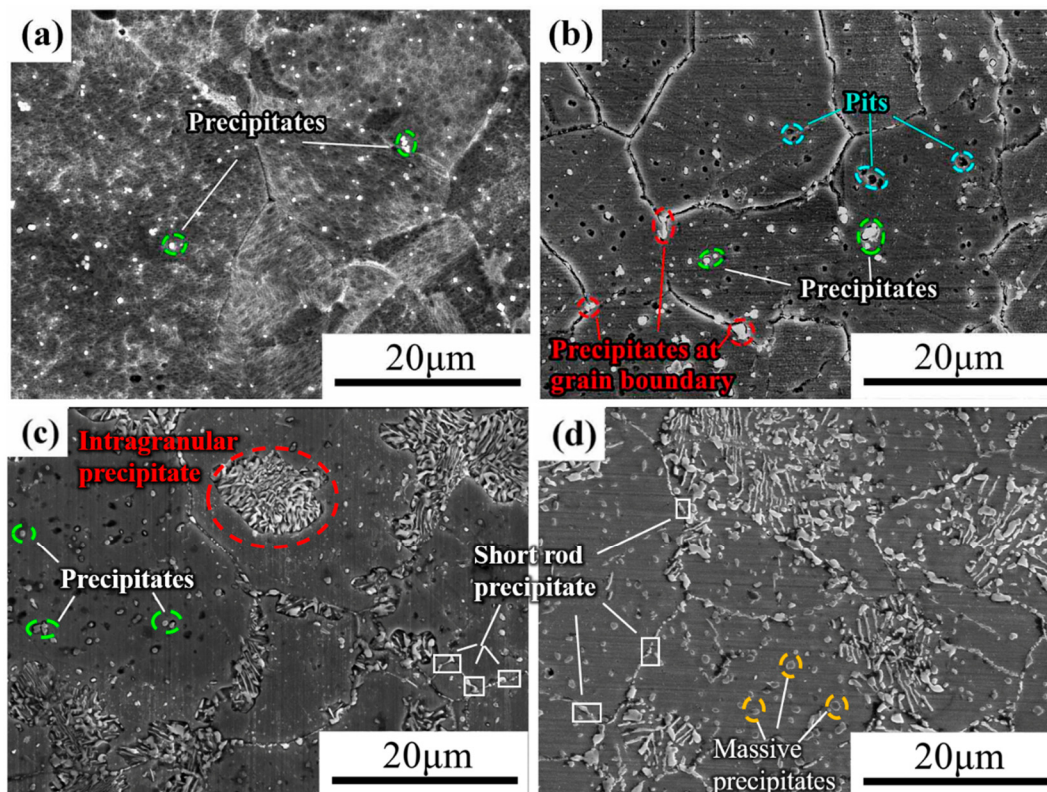


Figure 4. Microscopic morphology of HNASS after 3 h of aging at different temperatures. (a) 600 °C: dispersively distributed granular precipitates; (b) 800 °C: the size of the precipitates increased, and some of them fell off; (c) 900 °C: lamellar precipitates appeared; (d) 1000 °C: the lamellar precipitate content and the whole precipitate content increased.

After aging at 600 °C for 3 h, no second phase was found at the austenite grain boundaries, and fine precipitated phases were evenly distributed inside the austenite grains (Figure 4a). After a 3 h of aging at 800 °C, the austenite grain boundaries become more visible, while a small amount of granular precipitates can be observed at some grain boundaries. The particles of the precipitated phase inside the crystal grains are larger than those for 600 °C, and part of the precipitates are found to fall off, with pits remaining on the austenite matrix (Figure 4b). The microstructure of the sample after aging at 900 °C for 3 h is shown in Figure 4c, where in discontinuous short rod-like precipitates appear at the austenite grain boundaries. The cell-like precipitates composed of lamellar precipitates and lamellar gaps are identified. The lamellar precipitates grow from the grain boundaries to the interior of the grain, and the lamellae direction and the grain boundaries are distributed at an acute angle. The granular precipitates could be observed in the austenite matrix, the size of which is not significantly different from that at 800 °C, and there is no obvious shedding. Furthermore, a small amount of large precipitates is also found on the matrix. It is more likely that the grain boundaries after aging at 1000 °C are occupied with short

rod-like precipitates, and the spacing of the lamellar precipitates inside the grains is slightly larger than for 900 °C. The granular precipitations inside the crystal grains did not change significantly as compared to that developed at 900 °C, but the massive precipitation phase increased significantly. With an increase in the aging temperature, the content of the precipitated phase also gradually increased, i.e., 0.8% → 5% → 12% → 17%.

3.3.2. Precipitated Phase after 5 h of Aging

At the same temperature, the precipitated phases in HNASS with different aging times display substantial differences. Figure 5 displays the SEM microstructural image of HNASS after aging at 800 °C for 5 h and demonstrates the micromorphology near the coarse grains. As compared to that after aging at 800 °C for 3 h in Figure 4b, more precipitates with a size of about 1 µm are detected in the substrate. Fine precipitates at the grain boundaries and the formation of lamellar precipitates could be observed around the part of the grain boundaries. At the boundaries of smaller grains (Figure 5b), a large number of short rod-like precipitates with a width of 1 µm and a length of about 2~4 µm as well as the lamellar precipitates are present. Increasing aging time from 3 to 5 h results in an improvement in the content of the overall precipitated phase from 5 to 11%. EDS analysis on the granular precipitates inside the grains and the short rod-like precipitates at the grain boundaries (Figure 5c,d) displays that the precipitates inside the grains contain V, while the precipitates at the grain boundaries are Vanadium-free. This potentially suggests that these are the two different types of precipitations.

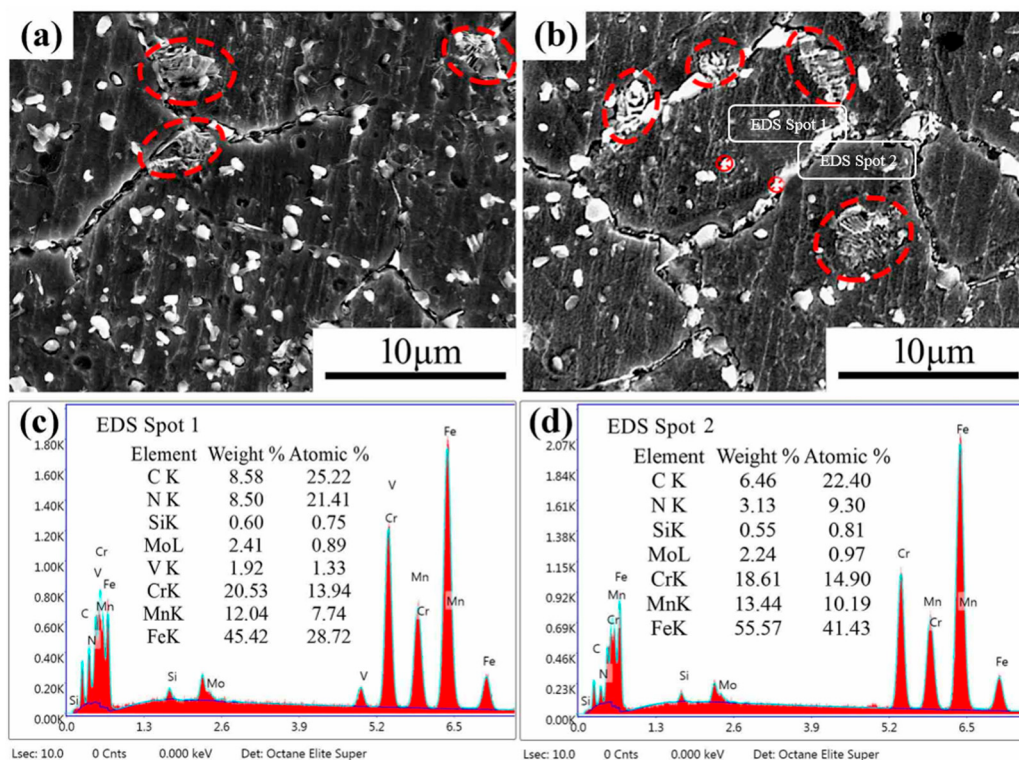


Figure 5. SEM micrograph and EDS analysis of HNASS after aging at 800 °C for 5 h. (a,b) SEM image: lamellar precipitates germinated at grain boundary; (c) EDS Spot 1: containing elements of V; (d) EDS Spot 2: no Element V, indicating that it is not the same precipitate as Spot 1.

Figures 6 and 7 exhibit the SEM microstructural image of HNASS after aging at 900 °C for 5 h and confirms the existence for four different shapes of precipitated phases. The lamellar precipitates, which grow from the grain boundaries to the inside of the grains, show lamellar space and the lamellar widths in the range of 100~300 nm and 70~100 nm, respectively. The short rod-like precipitates with a width of approximately

0.5 μm are present at the grain boundaries, while the massive precipitates with a size of about 0.5~1 μm are located inside the crystal grains. Additionally, the granular precipitates with a size of about 200 nm are dispersed on the austenite matrix. When the aging time was increased from 3 to 5 h, the content of precipitates increased from 12 to 22%. The morphology of the short rod-like precipitates at the grain boundaries was evaluated from discontinuous (Figure 4c) to end-to-end connected, and other precipitated phases did not change significantly. Figure 6c shows the results of EDS analysis of large precipitate (Spot 1), short rod-like precipitate (Spot 2) and lamellar precipitate (Spot 3) after aging at 900 $^{\circ}\text{C}$ for 5 h. Nitrogen is found exclusively in the lamellar precipitate (Spot 3), which is more likely the M_2X -type Cr_2N phase, according to the lamellar characteristics base on morphological analysis. The rod-like precipitate and lamellar precipitate are identified to be intermetallic compounds since the energy spectra of Spot 1 and Spot 2 are mainly composed of Fe, Cr, Mn and Mo elements.

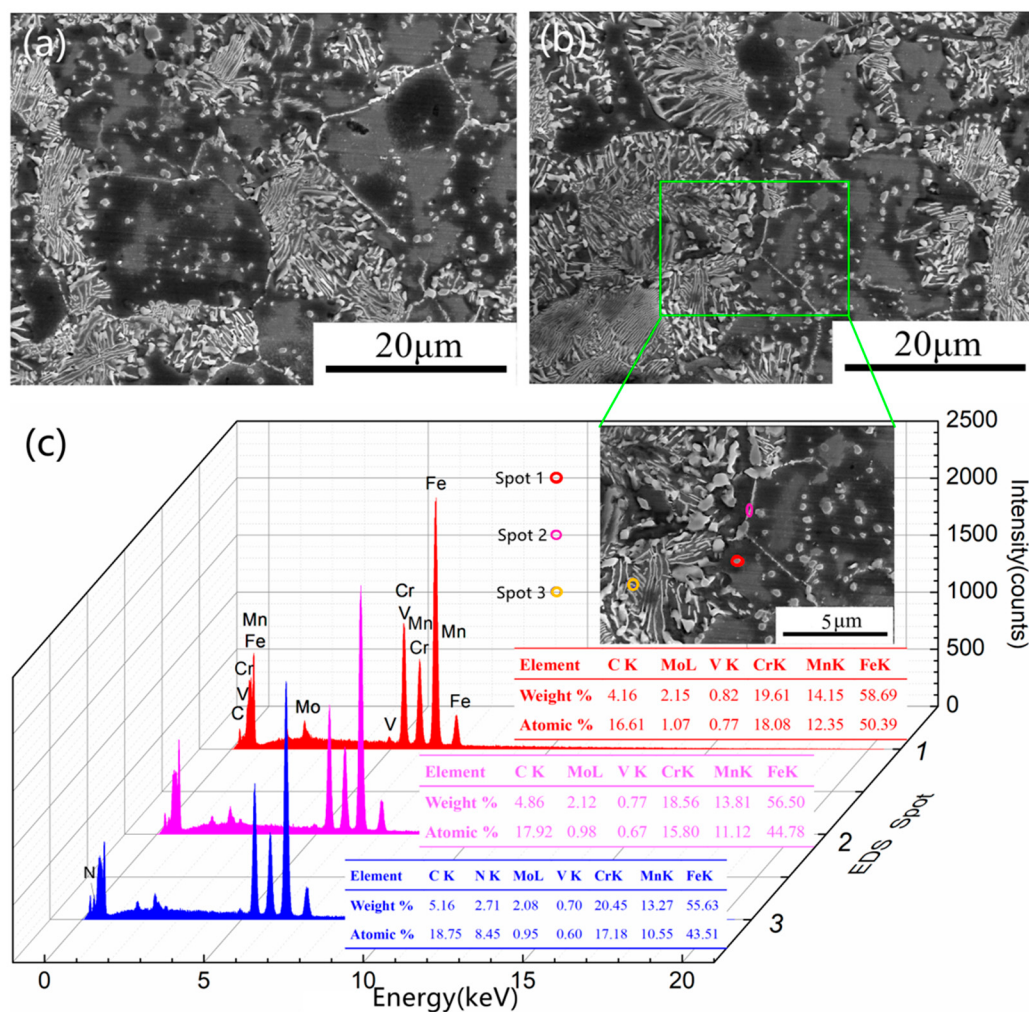


Figure 6. SEM micrographs of HNASS after aging at 900 $^{\circ}\text{C}$ for 5 h (a,b): compared with 3 h, the precipitate content increased, and EDS analysis (c): it is speculated that the precipitates at point 1 are Cr_2N , and the precipitates at point 2 and 3 are intermetallic phase.

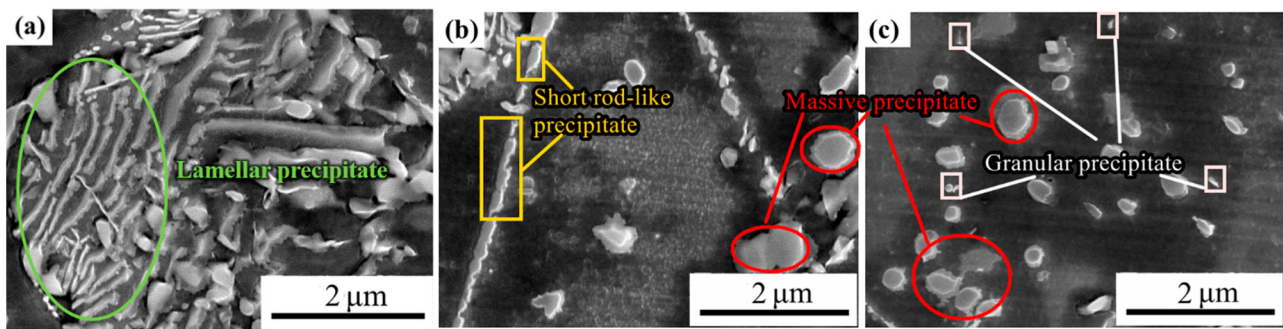


Figure 7. SEM morphology of the typical precipitates of HNASS after aging at 900 °C for 5 h. (a) Lamellar precipitate; (b) Short rod-like precipitate and Massive precipitate; (c) Granular precipitate and Massive precipitate.

4. Discussion

The excellent mechanical property of HNASS is associated with the presence of an interstitial solid solution of N in the steel. The occurrence of precipitation behavior leads to the depletion of N in the HNASS matrix, which reduces the material processing as well as mechanical properties. A deeper understanding of the influence of the addition of V and Nb on the precipitation behavior and the precipitation mechanism has obvious positive significance for effectively avoiding the degradation performance during the processing and utilization of HNASS [26].

4.1. Characteristics and the Mechanism of Formation of Precipitates

4.1.1. MX Phase

The MX phase is present in the original state and solid solution state, as well as aging state of HNASS. The MX phase is a non-stoichiometric carbonitride formed by the combination of microalloying elements of Nb, V, C and N. In general, its crystal structure is cubic with the chemical formula $(\text{Nb}_x\text{V}_{1-x})(\text{C}_y\text{N}_{1-y})$. The common MX phase is granular, which is dispersed into steel and has significant fine grain strengthening and precipitation strengthening effects [27]. An obvious difference in the sizes of MX phase particles could be noted, which is due to the ratio in the contents of Nb and V in the MX phase. Xu et al. [28] found a clear positive relationship between the size of the MX phase and $c(\text{Nb})/c(\text{V})$. Only the MX phase of large particles has a Nb(CN) core. In contrast, the small particles do not possess a core but have carbonitride solid solutions of Nb and V. The correctness of this model needs to be verified, but it is useful to understand the reasons for the observed differences in the size of the MX phase [29].

Studies have shown that the addition of V, Nb and other elements to HNASS can promote the formation of the MX phase, where Nb effectively improves the high-temperature stability of the MX phase [30]. Therefore, the MX phase is not completely dissolved after treatment with the solid solution and water cooling [31,32]. Due to the existence of the MX phase, solute drag and precipitated phase pinning effects occur during the hot deformation of HNASS, thereby suppressing the recrystallization of austenite and playing the role of refining the grains. High-temperature rolling and other mechanical processing methods could make the size of the MX phase significantly smaller, and the distribution conditions also change accordingly, which further improves the mechanical properties of the material [33].

4.1.2. M_2X Phase

A large number of lamellar precipitates appearing in HNASS could be noted after aging at 900–1000 °C for 3–5 h, as shown in Figure 4c,d and Figure 6a,b. This lamellar “nitrogen-containing pearlite” is a typical M_2X -type precipitated phase. In HNASS, the M_2X phase mainly stands for Cr_2N . The typical mechanism of precipitation of Cr_2N is the discontinuous cellular precipitation [34,35], which usually grows along the grain bound-

aries into the grains (Figure 8a), obeying the crystallographic OR: $[110]_{\gamma} // [1100]_{\text{Cr}_2\text{N}}$ and $(111)_{\gamma} // (0001)_{\text{Cr}_2\text{N}}$ [12]. The micromorphology of Cr_2N is affected by the aging temperature and time, which result in changes in the sheet thickness and sheet spacing [36]. As shown in Figure 8b, the growth direction of Cr_2N lamellae is not consistent within the same austenite grain; the Cr_2N lamellae on the same extension line are also discontinuous, along with the presence of granular precipitates at the discontinuities. Due to many defects and high energy at the grain boundaries, N atoms are easily captured, and Cr_2N is preferentially generated at the grain boundaries and grows into the grains [37,38]. At the same time, the austenite grain boundaries accompanied by the growth of Cr_2N also migrate. Knutsen et al. [26] supposed that the migration of austenite grain boundaries led to discontinuous cellular precipitation behavior of Cr_2N , and the key reason for the migration of grain boundary is the difference in the chemical potential between the transformed and unconverted austenite. However, more subsequent studies have shown that the discontinuous cellular precipitation behavior of Cr_2N is related to the short-range diffusion of Cr and the long-range diffusion of N from the untransformed austenite to unbalanced austenite. This study supports the theory of elemental diffusion. First, not only the growth of Cr_2N from the austenite grain boundaries but also the growth of Cr_2N in the austenite grains was observed, as shown in Figures 4c and 5b. This proves that without the migration of austenite grain boundaries, the discontinuous cell precipitation behavior of Cr_2N can still occur. Secondly, the longitudinal growth of flaky Cr_2N (growth on the same extension line) was not completely continuous.

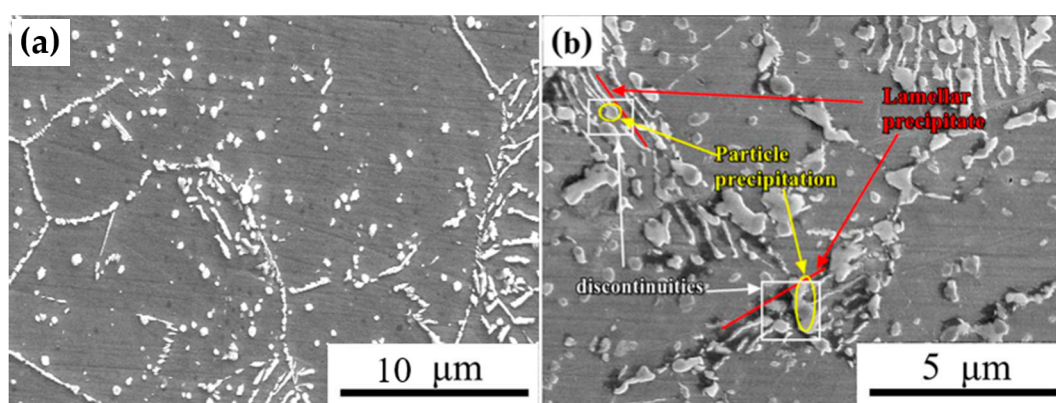


Figure 8. SEM morphology of HNASS at 1000 °C for 0.5 h (a) and SEM morphology of precipitates after 3 h (b).

The discontinuous Cr_2N lamellae grow from the granular precipitate and stop before the next particulate matter. The previous analysis proves that the granular precipitate is a large amount of MX phase and intermetallic phase with a high content of Cr. Studies [39,40] have shown that N diffuses from the γ phase and Cr diffuses from the intermetallic phase, forming Cr_2N at the interface between the γ and intermetallic phases. By considering the short-range diffusion of Cr, the granular precipitated phase can provide the corresponding interfacial energy. It can be speculated that the mechanism of discontinuous cell precipitation of Cr_2N is divided into two steps. First, the interface of austenite and other precipitated phases assists short-range diffusion of Cr to provide the basic conditions for the nucleation of Cr_2N . Then, the long-range diffusion of N from the untransformed austenite to austenite promotes the growth of discontinuous cellular precipitation of Cr_2N (Figure 9). The addition of microalloying elements such as V and Nb to HNASS improves the stability of MX phase and the equivalent intermetallic Laves phase, providing favorable conditions for the precipitation of Cr_2N phase. At the same time, it also supports the observations of Knutsen [26] that the V element can improve the stability of M_2X -type precipitates.

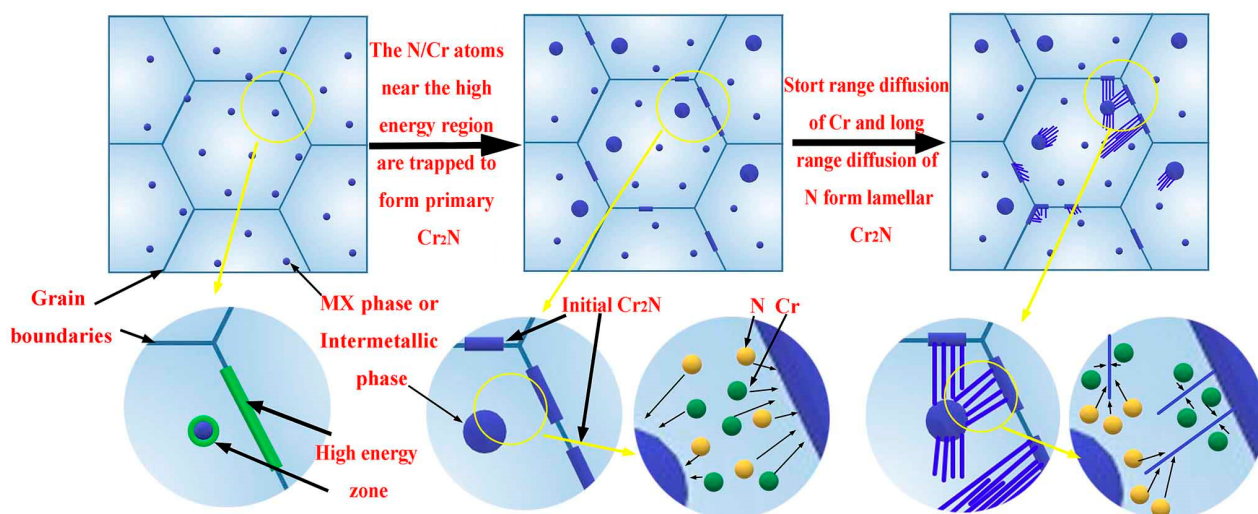


Figure 9. Schematic diagram of the formation mechanism of Cr_2N : at the early stage, Cr and N aggregated at the high-energy interface to form Cr_2N , and then Cr_2N grew from the interface to the grain in a certain orientation under the action of the long-range diffusion of N and the short range diffusion of Cr.

4.1.3. Intermetallic Phase

The σ , χ and Laves phases are the common precipitated phases in HNASS. The σ phase usually appears in the as-cast HNASS while absence in the aging HNASS. High N alloying can effectively suppress the formation of the σ phase. Since the N content in HNASS is as high as 1.021% and the aging treatment is performed for a maximum of 5 h in the heat treatment, the χ phase is an intermetallic phase of α -Mn type body-centered cubic structure which commonly exists in stainless steels. The temperature range of precipitation is narrow, and the precipitation time is long. It is often replaced by the M_2N phase. The existence of the χ phase cannot be determined in this study. The Laves phase is a hexagonal C14 or C36 type intermetallic phase with the chemical formula, $(\text{Fe}_{0.85}\text{Cr}_{0.15})(\text{Mo}_{0.3}\text{Nb}_{0.4}\text{Si}_{0.25}\text{Ti}_{0.05})$ and has excellent high-temperature mechanical properties. The addition of Nb to HNASS can promote the formation of Laves phase, and V can improve the stability of Laves phase [41]. The existence of MX phase leads to a reduction in the surrounding C and N elements and also contributes to the formation of the Laves phase [42]. Since both the MX and Laves phases are granular precipitates, below $5\ \mu\text{m}$ in size, it is not easy to distinguish them in OM and SEM photographs. Based on EDS (Figure 6), it is determined that the Laves phase is present in the granular precipitates of aging HNASS. The confirmation of the Laves phase needs to be further verified by more TEM observations and SAED calibration. The addition of V and Nb causes the formation of granular MX and Laves phases in HNASS, which indirectly promotes and stabilizes the precipitation of M_2X phase.

5. Conclusions

In this study, the precipitation behavior of the second phase in HNASS was investigated in terms of the heat treatment response. The following conclusions could be drawn:

(1) Solution/aging treatment experiments show that the MX phase, which exists in the as-produced HNASS, is not completely dissolved after a heat treatment at $1200\ ^\circ\text{C}$ for 1 h. Isothermal aging that was performed in temperature range $800\text{--}1000\ ^\circ\text{C}$ for 3 h induces the participation of M_2X phases. The content of the precipitates increases with increasing both the aging temperature and the aging time.

(2) The MX phase was precipitated by adding V and Nb in HNASS, which does not dissolve after solution and aging treatments. The microscopic morphology of the MX phase is granular, dominated by VN with a cubic structure and a lattice parameter of $a = 0.413\ \text{nm}$.

(3) M_2X is the main precipitation phase in HNASS treated by isothermal aging, and its typical representative is Cr_2N . The Cr_2N is present in the lamellar structure, where the thickness and spacing depend on the aging temperature and duration. The existence of MX phase provides interfacial energy for the precipitation of Cr_2N , such that Cr_2N nucleates in the austenite grain based on the interface of MX phase and extends toward the interior of the austenite.

Author Contributions: R.Z.; the determination of the framework of the article, the analysis of the experimental data and the modification of the content. W.S.; the expansion of the experiment and the acquisition of experimental data. C.Y.; the development and conduct of the experiment. X.Z.; the development and conduct of the experiment. Q.L.; perfect processing and content analysis of the experiment. Y.Z.; make the experimental plan and optimize the experimental process. L.C.; optimization and communication of experiments. All authors have read and agreed to the published version of the manuscript.

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