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TEM investigations of MN nitride phases in a 9% chromium ferritic/martensitic steel with normalization conditions for nuclear reactors

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ABSTRACT

The investigations on the precipitate phases in a 9%Cr ferritic/martensitic steel under different normalization conditions have been made by using a transmission electron microscope and an energy-dispersive X-ray spectroscopy. Hot-rolled steel samples were normalized at 1050–1200 °C for 1–2 h followed by an air cooling to room temperature. MN vanadium nitride precipitates with a plate-like morphology and a chemical formula of about $(V_{0.4}Nb_{0.4}Cr_{0.2})N$ have been observed at triple junctions, grain boundaries and within matrix in the steel samples normalized at 1050–1150 °C for 1–2 h, but they were dissolved out at 1200 °C within 1 h. Vanadium nitride is a stable phase at 1050 °C according to thermocalc prediction of equilibrium phases in the steel. With increasing normalizing temperature and time, there was no a striking change in the chemical composition of metallic elements in the MN phase, but a considerable increase in the size of the MN precipitate.

1. Introduction

As high temperature materials widely used in power plants and chemical industries, ferritic/martensitic steels with high strength and thermal conductivity, low thermal expansion, and good resistance to corrosion are being considered as candidate materials for application as fuel cladding or core structures of various generation IV nuclear reactors because of high resistance to radiation-induced swelling [1–4]. However, the creep rupture strength of ferritic/martensitic steels abruptly decreases during long-term creep exposure at high temperature [5]. At present, the creep strength of martensitic steels, which have been used for power-generation and chemical and petrochemical industries, is adequate to 550–600 °C [4]. To reach higher operating temperatures in advanced power plants, there has emerged a strong need to develop new steels for the applications at higher temperature. It has been made subject of ferritic/martensitic steels development for application at 650 °C [6,7]. The critical issues are the improvement of long-term creep strength as well as oxidation and corrosion resistance of ferritic/martensitic 9–12%Cr steels [8].

One of the major factors (or mechanisms) for improving the creep strength of metals and alloys is dispersion hardening. The creep resistance of martensitic steels is enhanced by a fine dispersion of non-shearable particles which are resistant to coarsening [9]. Formation of more stable and fine precipitation is key issue in improving the creep rupture strength of ferritic/martensitic steels. Previous studies have shown that in 9%Cr steel the volume fraction of VN precipitates remains constant during creep [10], and the VN precipitates are highly resistant to coarsening at both 600 and 650 °C, implying that this phase contributes equally to creep strength at the two temperatures [11]. The creep rupture strength of 9%Cr steel could be improved by a fine dispersion of vanadium nitride precipitates. Therefore, the investigation of vanadium nitride precipitates in 9%Cr steel is important to understand creep properties of the steel.

9–12% Chromium steels are commonly used in normalized and tempered condition. Normalizing is done at a temperature, where the steels are austenitic (usually at 1040–1100 °C). Normalizing dissolves the majority of nitrides, carbides or carbonitrides. As the solubility of VN at normalizing temperatures is significantly higher than that for Nb(C,N), complete dissolution of VN can be expected at relatively low temperatures. The only secondary phase staying undissolved at normalizing temperature is Nb(C,N). Tempering is carried out in the range 650–780 °C. Precipitation of fine VN and/or Nb(C,N) takes place during tempering due to lower solubility of those phases in ferrite. Moreover, precipitation of $M_{23}C_6$ and M_2N (mainly Cr_2N) takes place, too. Normalizing and tempering heat treatment leads to a microstructure of tempered martensite consisting of fine ferrite subgrain with $M_{23}C_6$ carbides precipitated on subgrain boundaries and on prior austenite grain boundaries, and fine MX carbonitrides distributed uniformly within subgrains or on subgrain boundaries, as well as M_2N nitrides within subgrains [12–16].

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Only a little publication regarding to precipitation of vanadium nitride in 9%Cr steels with a normalizing condition has been found [17]. In this paper, we reported the vanadium nitride precipitate phase observed in a 9%Cr steel under different normalization heat treatment conditions.

2. Experimental

A 9% chromium steel plate of 15 mm in thickness, prepared by melting, casting and hot rolling (at 1150 °C) was used in this study. The chemical composition of the steel was <0.003C–9.88Cr–0.51Mo–2.09W–0.20V–0.20Nb–0.43Mn–0.37Ni–0.105Si–0.084N (in wt%). The heat treatments performed on hot-rolled steel samples were normalization at 1050 °C for 1 h, 1100 °C for 1 and 2 h, 1150 °C for 2 h and 1200 °C for 1 h followed by air cooling (AC) to room temperature, named after S1, S2, S3, S4 and S5, respectively.

Extraction carbon replicas on copper grids were used for precipitate analysis. For preparation of the replicas, normalized steel samples were first mechanical ground and polished, and then chemical etched in a solution of 2%HF–5%HNO_3 in distilled water. After evaporating carbon onto the etched surface of the sample, the scratched carbon films were floated to the surface using a solution of 10%HCl in methanol and an applied voltage of 2 V at room temperature.

The examination of extraction carbon replica was performed on a JEM-2000 FXII TEM (transmission electron microscope) equipped with a LINK EDX (energy-dispersive X-ray)-system and operating at 200 kV accelerating voltage.

3. Results and discussion

Fig. 1(a) and (c) shows the TEM micrograph of S1 steel sample, normalized at 1050 °C for 1 h, taken from extraction carbon

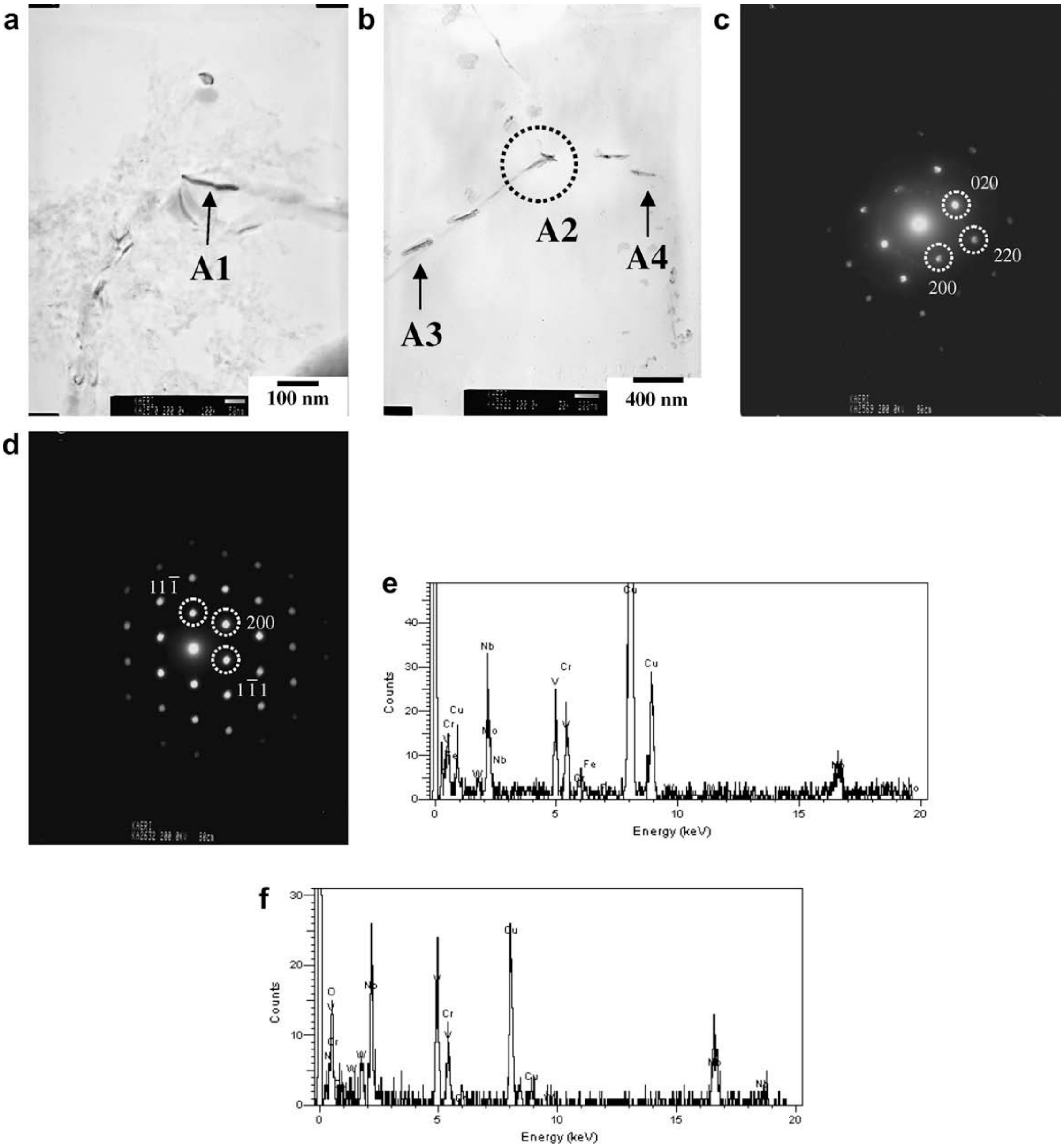

Fig. 1. (a) and (b) TEM extraction replica micrographs of 9%Cr steel normalization treated at 1050 °C for 1 h, showing vanadium nitride precipitates A1–A4. (c) and (d) microdiffraction patterns from precipitates A1 and A2 in the beam directions of [001] and [011], respectively. (e) and (f) EDX spectra from precipitate A1 and A2, respectively.

replica and microdiffraction (MD) pattern recorded from precipitate A1, respectively. Fig. 1(e) gives EDX spectrum from precipitate A1. The composition of metal fraction in precipitate A1 is listed in Table 1. The EDX analysis indicated that precipitate A1 is a V-Nb-rich nitride phase containing Cr. The MD pattern completely coincide with the electron diffraction pattern from VN (JCPDS file 35-0768) in the beam direction of [001]. Therefore, precipitate A1 was determined as vanadium nitride phase containing Nb and Cr with a face centred cubic (f.c.c.) crystal structure and a chemical formula approximately expressed as $(V_{0.35}Nb_{0.35}Cr_{0.30})N$.

Fig. 1(b) is another TEM micrograph taken from extraction carbon replica of S1 steel sample. MD pattern from precipitate A2, Fig. 1(d), matched well with the diffraction pattern from VN (JCPDS file 35-0768) in the beam direction of [011]. EDX spectrum and chemical composition with regard to precipitate A2 are given in Fig. 1(f) and Table 1, further indicating that precipitate A2 is vanadium nitride phase containing Nb and Cr. Precipitates A3 and A4, as shown in Fig. 1(b), with chemical compositions listed in Table 1 similar to that of precipitate A1, are also vanadium nitride phase containing Nb and Cr.

Microstructural studies on the precipitation behavior during normalizing heat treatment in creep resistant 9Cr1MoVNb steel, where the chemical composition of the steel was 0.09C–8.70Cr–0.90Mo–0.22V–0.072Nb–0.35Mn–0.28Ni–0.29Si–0.032Cu–0.001Al–0.009P–0.002S–0.044N (wt%), indicated that no V-rich MX is detected in the as normalized condition (1050 °C/10 min/AC), precipitation of V-rich MX occurs during tempering [14]. Also, there is no vanadium nitride in X20CrMoV121 steel, where the chemical composition of the steel was 0.20C–11.59Cr–0.98Mo–0.28V–0.47Mn–0.39Ni–0.24Si–0.026P–0.009S–0.032N (wt%), after normalization at 1050 °C for 1 h followed by AC [17]. On the contrary, V-rich MX containing Nb and Cr was observed in P91 steel with a chemical composition of 0.10C–8.30Cr–0.95Mo–0.21V–0.37Mn–0.07Nb–0.15Ni–0.36Si–0.011P–0.001S–0.053N (wt%) after a normalization heat treatment (1050 °C/1 h/AC) [17], in line with our TEM observation regarding to S1 steel sample. Contents of nitrogen and carbon can affect the precipitation behavior of MN nitrides during normalizing. With increasing content of nitrogen or carbon, the solubility at normalizing temperature such as 1100 °C for VN, Nb(C,N) in austenite with respect to a 11 wt%Cr steel decrease [12]. Therefore, the amount of MN nitrides should increase during normalizing with increasing the content of nitrogen or carbon in austenite of high Cr steels. It can be suggested that the reason why the different behavior appeared in the precipitation behavior of MN nitrides between the present steel and steels mentioned above is mainly caused by the different content of nitrogen in austenite in those steels.

Fig. 2(a), (b) and (c) shows TEM images of extraction carbon replica prepared from steel sample (i.e., S2) normalized at 1100 °C for 1 h followed by AC. Precipitates A5 and A6 were determined to be vanadium nitride containing Nb and Cr, according to EDX results (Fig. 2(f) and (g), respectively, and Table 1) and MD patterns Fig. 2(d) and (e), which matched well with those of VN (JCPDS file 35-0768) in the beam direction of [001] and [011], respectively. Precipitates A7–A9, where their chemical compositions are listed in Table 1, were also vanadium nitride containing Nb and Cr.

It has been reported that V-rich MX-type carbonitride precipitate phase could not be found in high Cr steels, where the chemical compositions of the steels are 0.082–0.12C, 9.16–9.28Cr, 3.3W, 3Co, 0.2V, 0.05Nb and 0.001–0.051N (wt%), after normalization at 1100 °C for 30 min followed by AC [13]. After normalization at 1100 °C for 1 h, many MX-type precipitates with niobium concentration of 40–70 mass% were observed in a 9Cr–1Mo–V–Nb steel with a chemical composition of 0.09C–8.93Cr–0.98Mo–0.22V–0.41Mn–0.067Nb–0.26Si–0.051N (wt%) [15]. It is not clear that those precipitates belong to vanadium nitride or niobium nitride. Our observed vanadium nitride precipitates have niobium concentration of 40–50 mass%.

Table 1

Compositions of metal fraction in the vanadium/or niobium nitride precipitate in 9%Cr steel samples with different normalization treatments.

Sample	Precipitate	Composition (at.%)					
		V	Cr	Fe	Nb	Mo	W
1050 °C 1 h (S1)	A1	36.06	27.01	1.10	33.77	0.15	1.92
	A2	41.03	14.49		39.80		4.67
	A3	41.83	22.81	2.16	30.38	1.43	1.39
	A4	38.94	28.27		31.54		1.25
1100 °C 1 h (S2)	A5	38.11	19.37	1.70	38.03		2.80
	A6	50.29	15.06	0.28	33.31		1.07
	A7	42.60	20.40	1.02	30.83		5.15
	A8	32.97	32.11	0.77	29.83		4.52
	A9	36.98	28.57	1.13	30.02		3.31
1100 °C 2 h (S3)	A10	39.10	18.91	2.07	39.02		0.92
	A11	47.84	19.35	1.64	30.35		0.81
	A12	38.02	20.79		39.29		1.91
	A13	40.43	17.27	0.30	40.84		1.17
1150 °C 2 h (S4)	A14	43.68	14.46		41.30		0.56
	A15	45.47	15.17	0.27	37.64		1.44
	A16	43.48	12.11		43.59		0.82
	A17	41.41	17.22	0.36	40.10		0.91
	A18	45.93	23.49		28.54		2.04
1200 °C 1 h (S5)	B1	23.81	16.16	1.98	57.49		0.57
	B2	27.90	15.83	2.28	53.04		0.94
	B3	21.65	14.07	0.93	62.49		0.85
	B4	22.23	10.07		67.18		0.52
	C1	21.10	16.47		62.42		
	C2	27.71	7.26		63.87		1.16
	C3	21.30	14.03	0.79	63.45		0.43
	C4	20.90	18.97	2.26	57.37		0.50
	C5	23.60	12.65	2.66	60.31		0.78

Precipitates A10 (Fig. 3(a)) observed in S3 steel sample normalized at 1100 °C for 2 h were also determined to be vanadium nitride phase, in terms of MD pattern (Fig. 3(b)) taken from precipitate A10, which matched with diffraction pattern of VN (JCPDS file 35-0768) in the beam direction of [011], and result of EDX analysis (Fig. 3(c) and Table 1), so did the precipitates A11–A13 as shown in Fig. 3(a). It seems that there is no obvious change in the chemical composition and size of vanadium nitride when increased normalization time.

In the S4 steel sample normalized at 1150 °C for 2 h, we also observed many vanadium nitride precipitates, A14–A18 as shown in Fig. 4(a) and (b). Fig. 4(c) is MD pattern recorded from precipitate

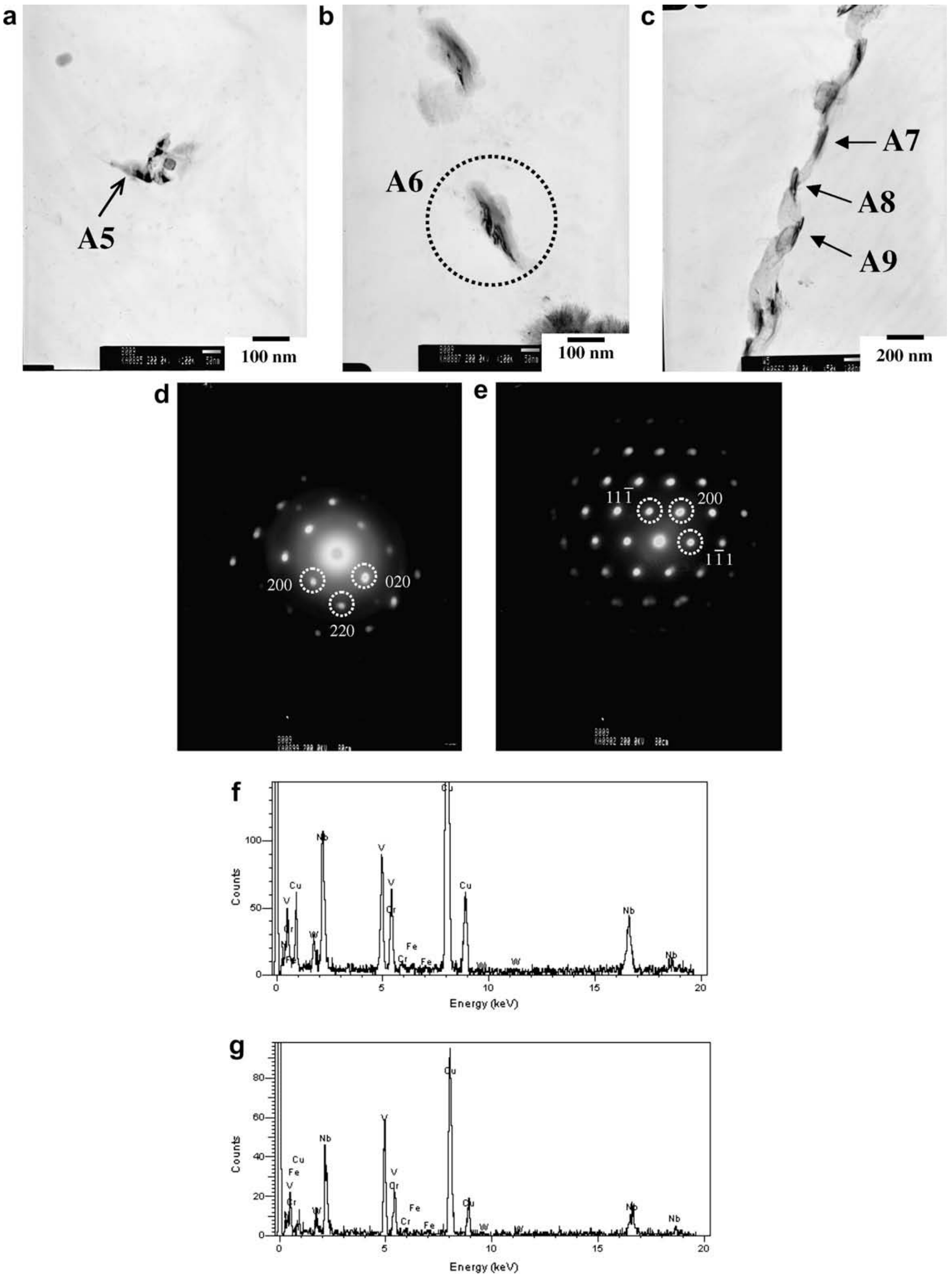

Fig. 2. (a), (b) and (c) TEM extraction replica micrographs of 9%Cr steel normalization treated at 1100 °C for 1 h, showing vanadium nitride precipitates A5–A9. (d) and (e) microdiffraction patterns from precipitate A5 and A6 in the beam directions of [001] and [011], respectively. (f) and (g) EDX spectra from precipitates A5 and A6, respectively.

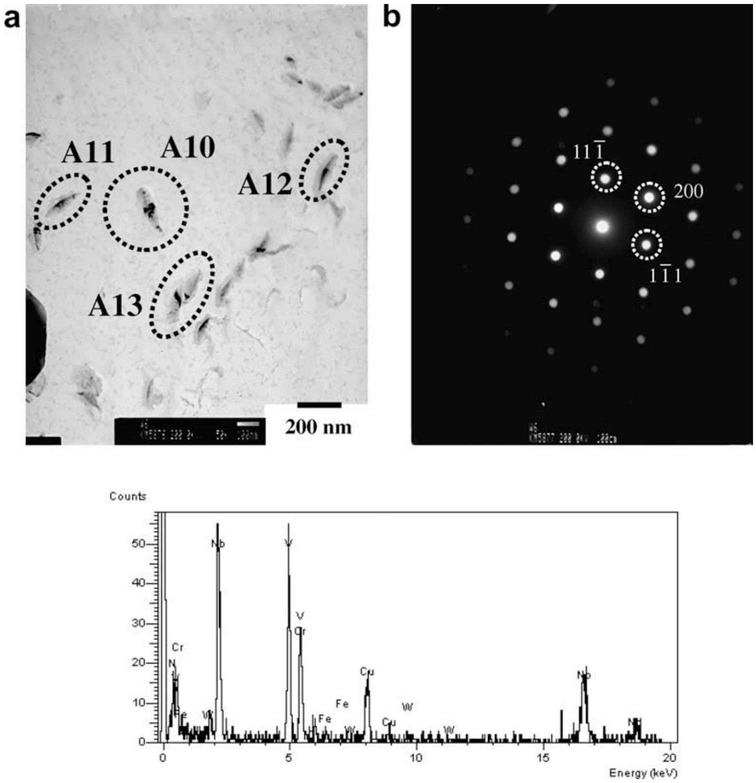

Fig. 3. (a) TEM extraction replica micrograph of 9%Cr steel normalization treated at 1100 °C for 2 h, showing vanadium nitride precipitates A10–A13. (b) microdiffraction pattern from precipitate A10 in the beam direction of [011]. (c) EDX spectrum from precipitate A10.

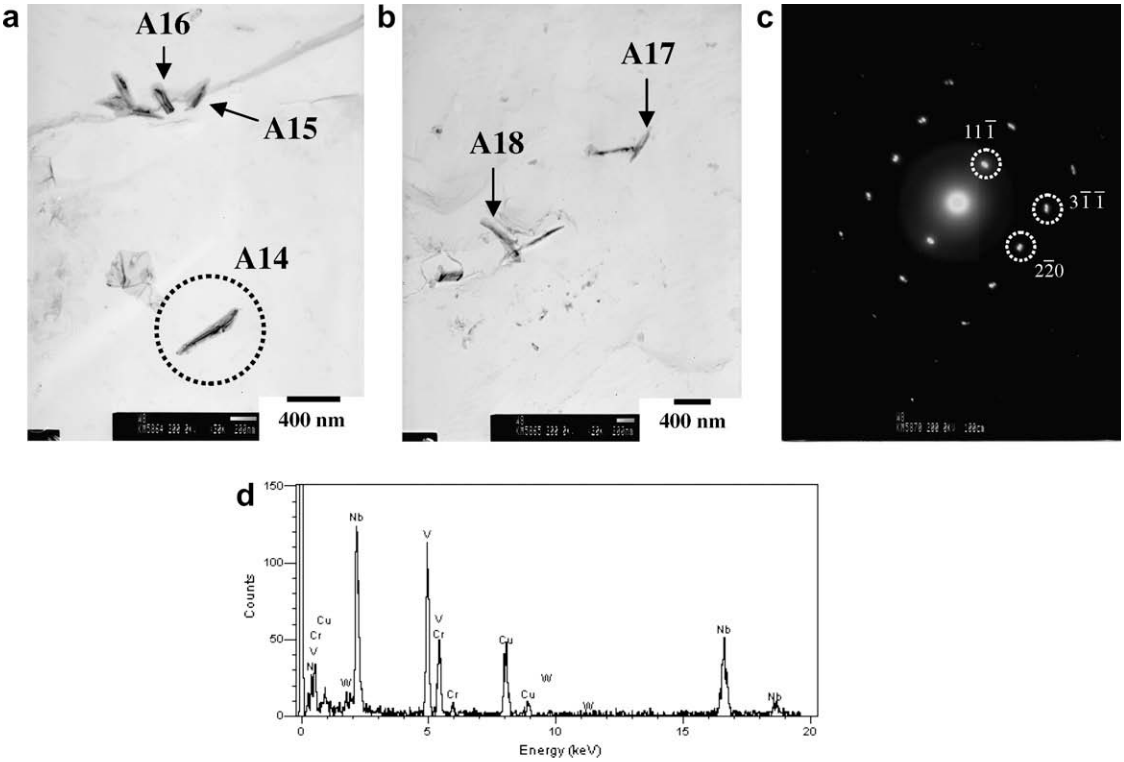

Fig. 4. (a) and (b) TEM extraction replica micrographs of 9%Cr steel normalization treated at 1150 °C for 2 h, showing vanadium nitride precipitates A14–A18. (c) microdiffraction pattern from precipitate A14 in the beam direction of [112]. (d) EDX spectrum from precipitate A14.

A14. This pattern was matched well with that of VN (JCPDS file 35-0768) in the beam direction of [012]. EDX spectrum from precipitate A14 and chemical composition of precipitates A14–A18 are given in Fig. 4(d) and Table 1, respectively. Present result is obviously different from that reported by Yoshino et al. [15] on an 9Cr steel (with a composition of 0.09C–8.93Cr–0.98Mo–0.22V–0.41Mn–0.067Nb–0.26Si–0.051N, in wt%) in the as normalized condition (1150 °C/10 min). They only observed small number of MX particles contained high niobium concentration of 80–95 mass% (i.e., NbX precipitate phase).

Fig. 5(a) and (b) shows TEM micrographs of carbon replica prepared from the S5 steel sample normalization treated at 1200 °C for 1 h, showing precipitates B1–B4 and C1–C5. It seems that plate-like precipitates B1–B4 and block-like precipitates C1–C5 are different phases, vanadium nitride and niobium nitride, respectively. However, EDX analyses (Table 1) indicated that these

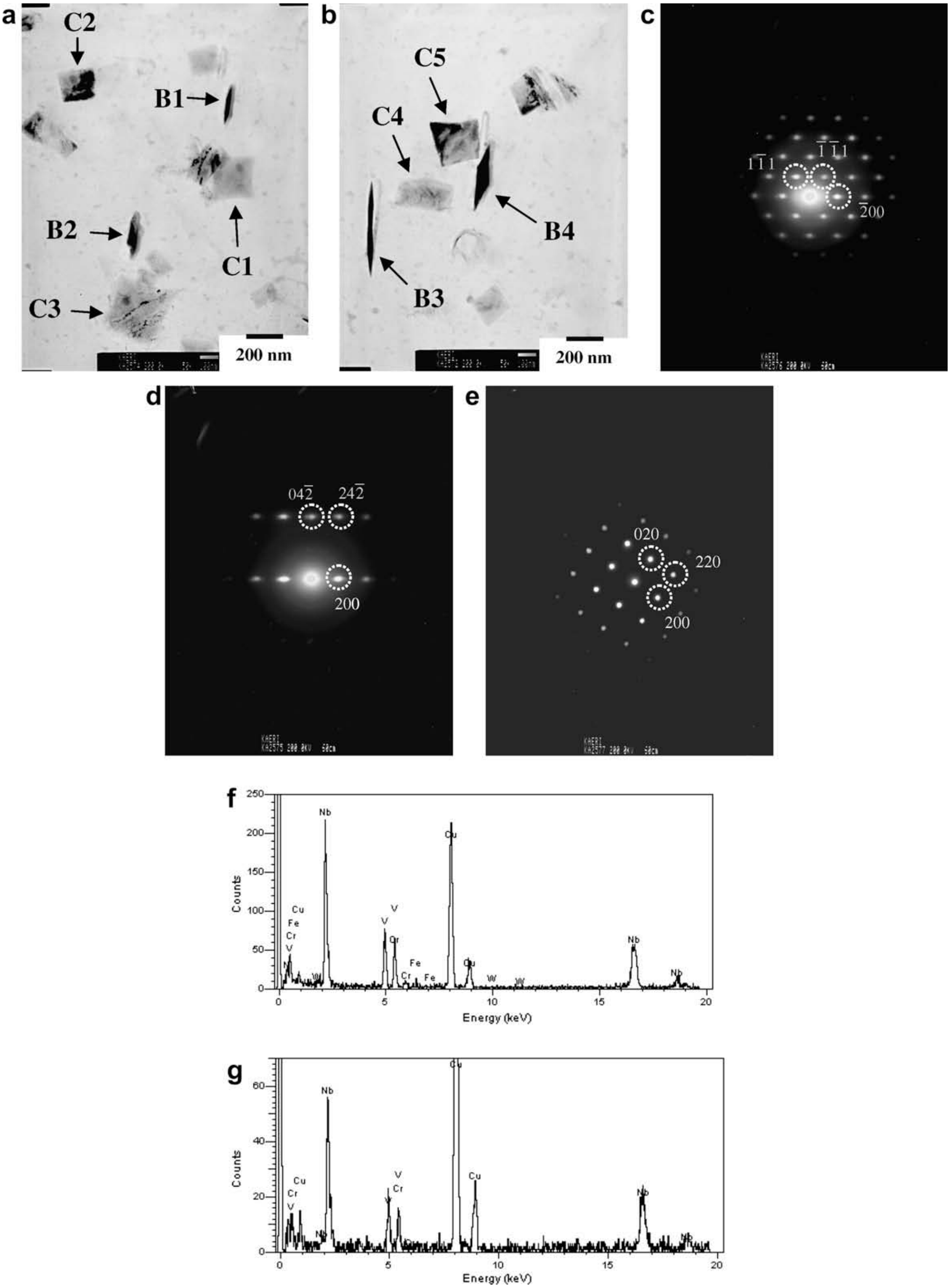

Fig. 5. (a) and (b) TEM extraction replica micrographs of 9%Cr steel normalization treated at 1200 °C for 1 h, showing niobium nitride precipitates B1–B4 and C1–C5. (c), (d) and (e) microdiffraction patterns from precipitates B1 and C1 in the beam directions of [011], [012] and [001], respectively. (f) and (g) EDX spectra from precipitates B1 and C1, respectively.

precipitates are nitrides with a similar chemical composition. Fig. 5(f) and (g) are EDX spectra from precipitates B1 and C1, respectively. Moreover, MD patterns shown in Fig. 5(c) and (d) taken from precipitate B1, as well as Fig. 5(e) taken from precipitate C1 matched well with those of NbN (JCPDS file 38-1155) in the beam directions of [0 1 1] and [0 1 2], as well as [0 0 1], respectively. Therefore, these precipitates belong to NbN phase containing V and Cr, and their chemical formula could be expressed approximately as $(Nb_{0.60}V_{0.25}Cr_{0.15})N$. It is noted that no vanadium nitride precipitate phase was detected in present 9%Cr steel normalized at 1200 °C for 1 h followed by AC. This result is in accord with the report that no vanadium nitride precipitate phase was detected in an 9Cr–1Mo–V–Nb steel under a normalization condition (1200 °C/1 h/AC) [15].

From the TEM observations for the replicas from the S1 to S5 steel samples, it seems that vanadium nitride is a stable phase at 1050–1150 °C for the present 9%Cr steel, whereas it is unstable and dissolved out at 1200 °C during the normalization treatment for 1 h. In order to clarify if above suggestion is reasonable, equilibrium phases in present 9%Cr steel have been predicted by using a Thermo-Calc program. The prediction result is shown in Fig. 6. From line 5 in Fig. 6 it is clear that VX is a stable phase at the temperature below 1070 °C, whilst it is unstable and should be dissolved at the temperature higher than 1070 °C. We examined a replica sample prepared from present hot-rolled steel sample. Vanadium nitride phase with a chemical formula approximately expressed as $(V_{0.45–0.53}Nb_{0.28–0.45}Cr_{0.10–0.20})N$ was observed in the sample. Thus, vanadium nitride existed in the S1 steel sample remained from the hot-rolled sample is stable phase and could not be dissolved during the normalizing at 1050 °C. In the steel samples normalized at 1100 and 1150 °C for 1–2 h, the presence of a vanadium nitride phase probably due to that the normalizing time is not long enough to completely dissolve out the nitride phase.

It has been suggested that the chemical compositions of metallic elements in MX carbonitride precipitate was strongly influenced by normalizing temperature [15]. The chemical composition of metallic elements in vanadium nitride in present normalized 9%Cr steel is about 0.35–0.50 V, 0.30–0.45Nb, 0.10–0.35Cr and 1–5W (at.%)/or 0.25–0.40V, 0.40–0.55Nb, 0.10–0.25Cr and 2–10W (wt%). The content of vanadium and niobium is slightly increased, whereas chromium content is decreased slightly with increasing normalizing temperature. In a general way, there is no large difference in chemical composition among these MX vanadium nitrides with increasing normalizing temperature from 1050 to 1150 °C. The chemical formula of vanadium nitride in S1, S2, S3 and S4 steel sample could be approximately expressed as $(V_{0.40}Nb_{0.35}Cr_{0.25})N$, $(V_{0.40}Nb_{0.35}Cr_{0.25})N$, $(V_{0.40}Nb_{0.40}Cr_{0.20})N$ and $(V_{0.45}Nb_{0.40}Cr_{0.15})N$, respectively. On the average, the chemical formula of vanadium nitride in the steel could be approximately expressed as $(V_{0.4}Nb_{0.4}Cr_{0.2})N$, which is strikingly different from that of vanadium nitride formed during a tempering at 750 °C for 2 h, as expressed as $(V_{0.6}Nb_{0.2}Cr_{0.2})N$ [18].

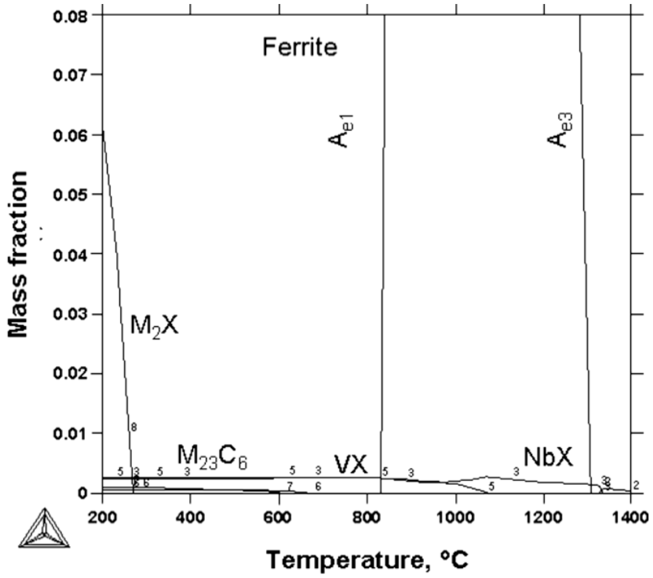

Fig. 6. Prediction of equilibrium phases in 9%Cr steel according to Thermo-Calc program. NbX, VX, $M_{23}C_6$ and M_2X phases are shown by lines 3, 5, 6 and 8, respectively.

The size in length of major axis of the plate-like vanadium nitride precipitate in the S1, S2, S3 and S4 steel samples was measured to be in the range of 50–250, 130–250, 150–300 and 240–650 nm and an average size of about 160, 185, 200 and 480 nm, which were from more than 10 measurements for individual vanadium nitride precipitate per sample, respectively. Thus, the size of MX vanadium nitride precipitate was affected by not only normalizing temperature but also normalizing time. This result agrees with the report that the particle diameter of MX precipitate was influenced by normalizing temperature [15].

For 9–12%Cr steels, the MX carbonitrides rich in V and Nb formed during tempering are distributed at laths, blocks, packet boundaries and prior austenite grain boundaries (PAGBs) as well as in the matrix within laths [19]. From present TEM observation of carbon replica of normalized steel sample, it can be seen that vanadium nitride phase distributed at PAGBs (Figs. 1(b), 2(c) and 4(a)) and triple junctions (for example, Fig. 1(b)). The distribution of many observed vanadium nitride particles from carbon replica sample cannot be determined. In order to determine the distribution thin foil sample should be used for TEM observation in our next experimental work. It can be expected that these vanadium nitride precipitates at grain boundaries are effective in preventing grain growth during normalization, and so that they contribute to improved toughness of the steel. On the other hand, since vanadium nitride precipitates are thermally stable and highly resistant to coarsening during creep exposure at 600–650 °C, pinning of the prior austenite and martensite lath boundaries can be occurred thus preventing recovery of the microstructure to take place.

It has been known that secondary fine MX phases formed during tempering and distributed throughout the matrix contribute to the creep strength of high Cr steel by acting as barriers to dislocation movement during high temperature deformation. Average sizes of vanadium nitride precipitates observed in the present normalized steel samples (S1–S5) are obviously larger than those formed during the tempering at 750 °C for 2 h, where the sizes in length of major axis of the plate-like vanadium nitride precipitates precipitated during the tempering are reported to be about 80–150 nm [18]. Thus, it is probably that the contribution to improving creep strength made by vanadium nitride precipitates in the normalized steel would be relatively small compared with that made by vanadium nitride precipitates formed during a tempering.

4. Conclusions

The investigations on the precipitate phases in a 9%Cr steel under different normalization heat treatment conditions have been made by TEM and EDX analysis. The investigations have lead to the following conclusions:

(1) Vanadium nitride precipitate phase containing Nb and Cr has been observed in 9%Cr steel samples normalized at 1050 °C for 1 h, 1100 °C for 1–2 h and 1150 °C for 2 h, but not in the sample normalized at 1200 °C for 1 h. Vanadium nitride is a stable phase at 1050 °C according to thermocalc

prediction of equilibrium phases in the steel. Present normalizing times at 1100 and 1150 °C are not long enough to completely dissolve the vanadium nitride, whereas the nitride can be dissolved out during a normalization at 1200 °C for 1 h.

(2) Vanadium nitride precipitate phase observed in the normalized 9%Cr steel has an approximate chemical formula of $(V_{0.4}Nb_{0.4}Cr_{0.2})N$, which is obviously different from that of vanadium nitride phase $((V_{0.6}Nb_{0.2}Cr_{0.2})N)$ formed during a tempering at 750 °C for 2 h.

(3) Normalizing temperature and time did not strongly affect the chemical composition of metallic elements in the MN vanadium nitride phase in the 9%Cr steel, whereas the size of MN vanadium nitride precipitate was considerably increased with increasing normalizing temperature and time.

(4) Vanadium nitride precipitates precipitated at triple junctions, grain boundaries, and within matrix. Some of the precipitates are effective in preventing grain growth during normalization and recovery of the microstructure, improving toughness and creep strength of the steel.

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