

# Vanadium nitride precipitate phase in a 9% chromium steel for nuclear power plant applications

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## Abstract

Vanadium nitride precipitate phase in a 9% Cr steel was observed and analyzed using transmission electron microscopy and energy dispersive spectroscopy. The steel samples were normalized at 1050 and 1100 °C for 1 h and then tempered at 750 °C for 30 min to 5 h followed by an air cooling. Through the microdiffraction pattern analyses and energy dispersive X-ray data, two kinds of vanadium nitride precipitates were determined to be  $(V_{0.6}Nb_{0.2}Cr_{0.2})N$  and  $(V_{0.45}Nb_{0.45}Cr_{0.1})N$  with the same fcc crystal structure and different lattice parameters  $\bar{a} = 4.070$  and  $4.232$  Å, respectively. Lattice parameters estimated for the precipitates regarding the VN phase agree well with the present data from the microdiffraction patterns, indicating that the precipitates do not belong to the VC phase. Observed  $(V_{0.45}Nb_{0.45}Cr_{0.1})N$  precipitates consisted of undissolved particles remaining after a normalizing and the particles newly precipitated during a tempering, whilst, the observed  $(V_{0.6}Nb_{0.2}Cr_{0.2})N$  precipitates were formed during a tempering. These two vanadium nitrides seem to be a stable phase, and not an intermediary phase.

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## 1. Introduction

High chromium ferritic–martensitic steels, such as 9–12% chromium steels, have been considered as a substitute for austenitic steels and potential material candidates in nuclear reactor systems to be used in the first wall and blanket structures of demonstration and commercial fusion reactors, for instance [1–3]. Primary reasons for the use of ferritic–martensitic steels are their higher resistance to an irradiation embrittlement and a radiation-induced swelling, also their higher thermal conductivity, and lower thermal expansion when compared to type 316 stainless steel and their superior creep resistance at a temperature up to about 620 °C [1,4–6].

The creep strength of metals and alloys is improved by three major factors, i.e., grain coarsening, solution hardening [7] and dispersion hardening [8], respectively. Conse-

quently, in the development of steel technology, much attention has been paid to the investigation of microstructure such as precipitate phase in steel.

The creep resistance of martensitic steels is enhanced by a fine dispersion of non-shearable particles which are resistant to a coarsening [9]. Vanadium nitrides are good in this context. It has been shown that plate-like V-nitrides are effective obstacles against a dislocation glide, and the length of the major axis of plate-like V-nitrides is considered to be one of the controlling factors of a creep resistant [10]. Meanwhile, previous studies have also shown that in 9% chromium steel the volume fraction of the VN precipitates remains constant during a creep [11], and the VN precipitates are highly resistant to a coarsening at both 600 and 650 °C, implying that this phase contributes equally to a creep strength at these two temperatures [12]. Moreover, it can be reasonable to assume that the fine vanadium nitride precipitates may play a significant role in improving the void swelling resistance after an irradiation like fine and stable dispersions of MC particles, which can act directly to

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impart a void swelling resistance [13]. Therefore, the investigation of vanadium nitride precipitates in high chromium ferritic–martensitic steels is very important to understand their creep properties and resistance to irradiation damage.

Vanadium nitride (VN) has been observed in 9–12% chromium steels [11,12,14–17]. Very little literature with regard to a vanadium-rich nitride precipitate MN containing chromium and niobium in 9–12% chromium steels was found [18–21].

In the present investigation, the vanadium nitride precipitates in developed 9% chromium steel with a normalized and tempered condition were studied by TEM (transmission electron microscope) and EDX (energy-dispersive X-ray) analysis.

## 2. Experimental details

The material used in this study was a 9% chromium steel prepared by a melting, casting and hot rolling to obtain a plate with a thickness of 15 mm. The chemical composition in weight pct of the steel was < 0.003 C, 9.88 Cr, 0.51 Mo, 2.09 W, 0.20 V, 0.20 Nb, 0.43 Mn, 0.37 Ni, 0.105 Si and 0.084 N. The heat treatments performed on the steel plate included the following: (1) normalizing at 1050 and 1100 °C for 1 h, air cooling; (2) tempering at 750 °C for 30 min to 5 h, air cooling.

The examination of the precipitate phases in the steel was performed on a JEM-2000 FXII TEM equipped with a LINK EDX-system and operating at a 200 kV accelerating voltage. Extraction carbon replicas on copper grids were used for the precipitate analysis to avoid matrix effects in obtaining EDX spectra. Precipitate phases were identified by a combination of an electron microdiffraction and an EDX analysis.

For the preparation of the carbon replicas, a steel sample was first mechanically ground and polished, and then chemically etched in a solution of 2%HF–5%HNO<sub>3</sub> in distilled water. After evaporating the carbon onto the etched surface of a 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 20 °C.

## 3. Results and discussion

Fig. 1 shows the TEM micrograph of the 9% chromium steel taken from the extraction carbon replica and the electron microdiffraction patterns as well as the EDX spectrum from the circle-marked precipitate A1. The EDX analysis from precipitate A1, as shown in Fig. 1(e), indicates that the precipitate is a vanadium-rich nitride phase containing chromium and niobium. Microdiffraction patterns recorded from precipitate A1, as presented in Fig. 1(b), (c) and (d), completely coincide with the electron diffraction patterns from the vanadium nitride (VN, JCPDS file 25-1252) in three beam directions of [001], [112] and [013], respectively. Therefore, precipitate A1 was deter-

mined to be a vanadium-rich MN-type phase with a face centred cubic (fcc) crystal structure.

Fig. 2 reveals several vanadium-rich MN precipitates in the steel with a plate-like morphology and a size of 15–40 and 80–150 nm for the length of the short and long axis of the precipitates, respectively.

In addition to the chemical composition of the MN precipitate in the steel, the precipitates marked B1–G1 with circles or arrows in Fig. 2 were also EDX analyzed and the compositions of them along with that of the precipitate A1 in Fig. 1 were averaged to be about 51V–19Cr–17Nb–9N (at.%), as given in Table 1, while the composition of the metal fraction in the precipitate was 56V–21Cr–19Nb (at.%). Thus, the chemical formula of the MN precipitate phase in the steel can be approximately expressed as (V<sub>0.6</sub>Nb<sub>0.2</sub>Cr<sub>0.2</sub>)N.

The chemical composition of the vanadium-rich nitride in a ferritic 11% chromium steel has been reported to be about 57V–15Cr–10Nb–15N (wt%) [18], so that the composition of the metal fraction in the nitride was about 72V–19Cr–7Nb (at.%) and the nitride could be approximately expressed as (V<sub>0.7</sub>Nb<sub>0.2</sub>Cr<sub>0.1</sub>)N, which is different from present result. Meanwhile, Orr and Woollard [19] investigated the VN precipitate phase in a 9%Cr 1% MoNbVN steel with a normalized and tempered (1050 °C + 750 °C) condition and indicated that the composition of the metal fraction in the VN phase was 55V–26Cr–3Nb–9Fe (wt%), i.e. 60.9V–28.2Cr–1.82Nb–9.08Fe (at.%), so that the VN phase can be roughly expressed as (V<sub>0.6</sub>Cr<sub>0.3</sub>Fe<sub>0.1</sub>)N. They also observed some precipitate particles with compositions approximately half way between VN and NbC, i.e., 40V–14Cr–39Nb–7Fe (wt%)/ or 49.1V–16.8Cr–26.3Nb–7.8Fe (at.%), so that the precipitate phase can be roughly expressed as (V<sub>0.5</sub>Nb<sub>0.25</sub>Cr<sub>0.15</sub>Fe<sub>0.1</sub>)N. These results reveal a large difference from the present result. Also, there is striking difference in the composition of VN precipitate between present data and the data from Ref. [20].

This difference may be attributed to the reason that the chemical composition of the steel, tempering temperature, and time can influence the chemical composition of the nitride metallic constituent, like the description for a carbide in a steel [22].

The present TEM work revealed a compositional behavior of the MN precipitate phase in the steel. Two types of MN phases were observed in the present 9% chromium steel: one was vanadium-rich containing chromium and niobium as mentioned above, while the second was vanadium–niobium-rich containing chromium.

Fig. 3(a) shows the TEM image of the steel taken from the replica sample in the same way as the above mentioned extraction carbon replica. EDX spectrum from the circle-marked precipitate A2 could be found in Fig. 3(e) indicating that the precipitate is a vanadium–niobium-rich nitride phase containing chromium. Also, microdiffraction patterns recorded from precipitate A2, as presented in Fig. 3(b), (c) and (d), are accordant with the electron diffraction patterns

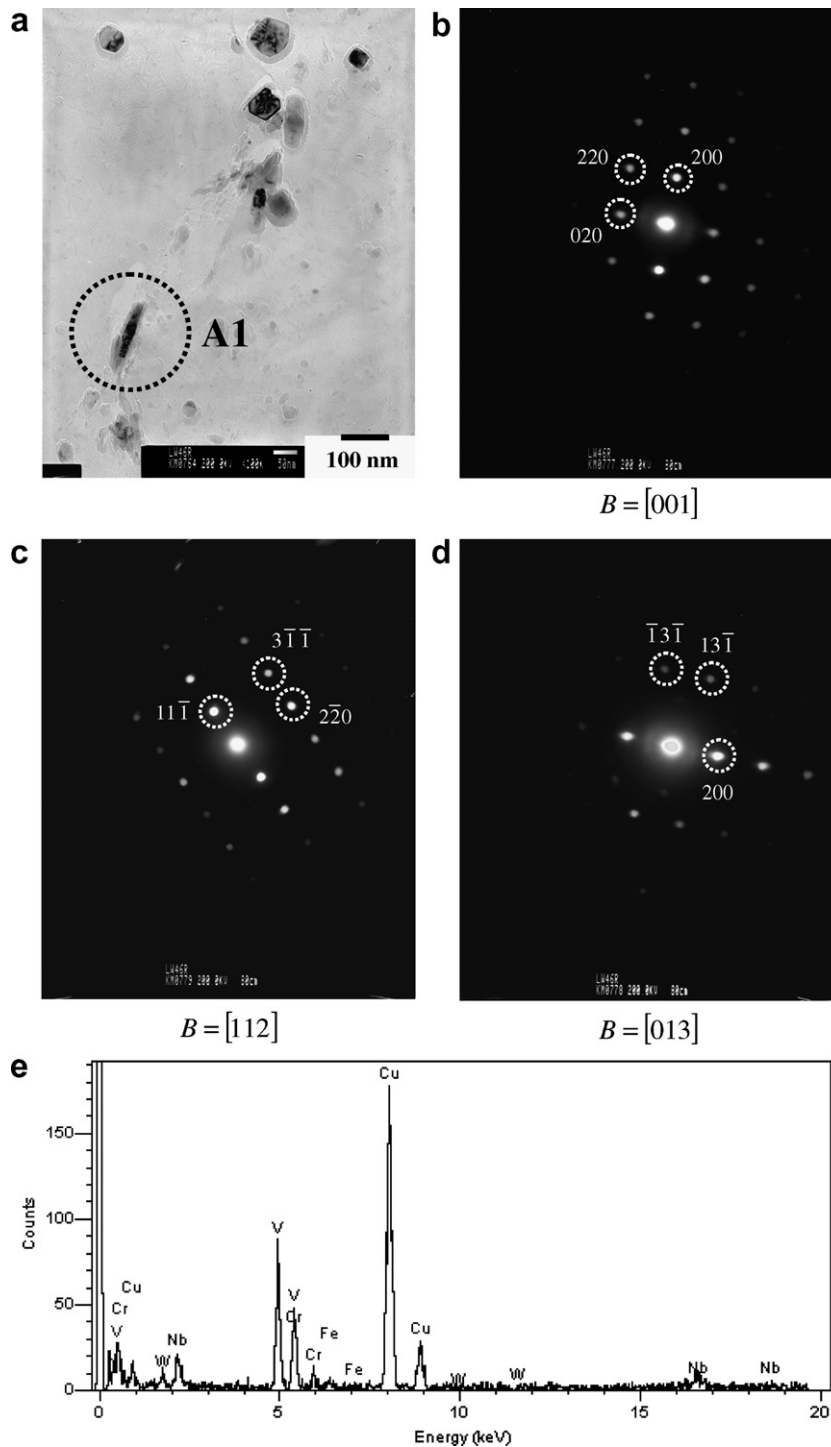


Fig. 1. (a) TEM extraction replica micrograph showing vanadium nitride precipitate A1 marked with circle; (b)–(d) microdiffraction patterns from precipitate A1 in three beam directions of  $[001]$ ,  $[112]$  and  $[013]$ , respectively; (e) EDX spectrum from precipitate A1.

from the VN (JCPDS file 25-1252) phase in three beam directions of  $[011]$ ,  $[012]$  and  $[125]$ , respectively. Thereby precipitate A2 was identified as a vanadium–niobium-rich MN phase with a fcc structure.

The vanadium–niobium rich MN precipitates have three kinds of morphologies: one was an equiaxial particle with a diameter of about 50 nm, while the second was a plate-like

shape with a size of about 20 and 120 nm in length of the short and long axis of the precipitates, respectively, and the third was rhombus with a size of about 100 and 150 nm in length of the short and long axis of precipitates respectively, as shown in Fig. 4.

The average chemical composition of the vanadium–niobium-rich MN phase from the precipitates A2–G2

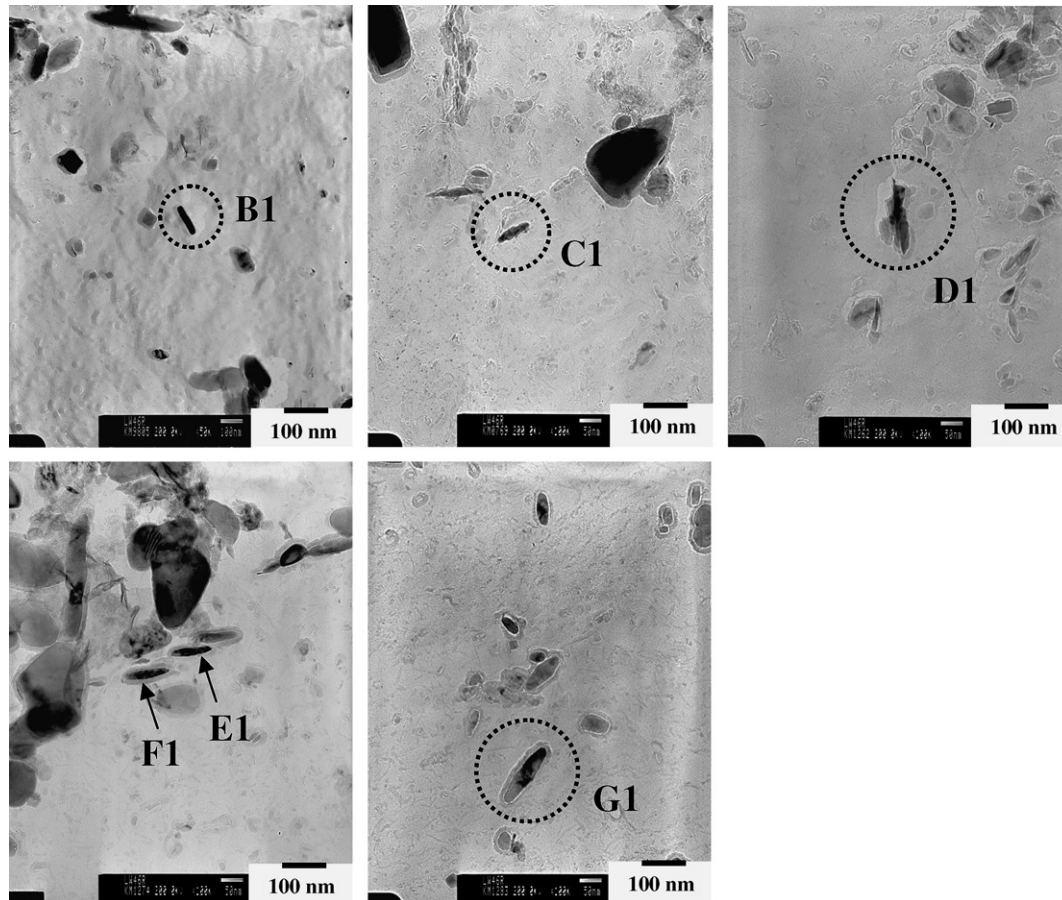


Fig. 2. TEM extraction replica micrographs showing vanadium nitride precipitates B1–G1 marked with circles or arrows.

Table 1  
Chemical compositions of the vanadium nitride precipitate phases in the 9% chromium steel

	Chemical composition (at.%)							Precipitate phase
	V	Cr	Fe	Nb	Mo	W	N	
A1	56.79	26.48	2.50	9.69		1.45	3.08	(V <sub>0.6</sub> Nb <sub>0.2</sub> Cr <sub>0.2</sub> )N
B1	59.85	14.68	0.36	17.80		1.08	6.23	
C1	44.63	24.36	1.86	13.98	3.39	1.35	10.43	
D1	52.10	26.98	2.52	8.62		1.18	8.60	
E1	51.38	19.14	0.91	21.33		0.97	6.27	
F1	50.82	13.17	0.71	19.19		0.66	15.45	
G1	44.54	12.31		29.63		1.56	11.97	
Average	51.44	19.59		17.18		1.18	8.86	(V <sub>0.45</sub> Nb <sub>0.45</sub> Cr <sub>0.1</sub> )N
A2	35.86	7.92	0.27	38.85		0.60	16.51	
B2	39.97	12.88		35.45		0.53	11.16	
C2	40.85	11.51		39.82		1.35	6.46	
D2	36.47	6.70	0.31	43.70		0.54	12.29	
E2	36.30	9.35	0.86	40.95		0.79	11.75	
F2	36.97	9.82	1.67	38.87		1.15	11.52	
G2	41.96	9.65	0.35	41.96		2.00	4.09	
Average	38.34	9.69		39.94		0.99	10.54	

shown in Fig. 4 were averaged to be about 38V–9Cr–39Nb–10N (at.%) as seen in Table 1 by EDX analysis, while the composition of the metal fraction in the precipitate was about 43V–11Cr–45Nb (at.%), so that the chemical formula of the vanadium–niobium-rich MN precipitate

phase in the steel can be approximately expressed as (V<sub>0.45</sub>Nb<sub>0.45</sub>Cr<sub>0.1</sub>)N.

Taneike et al. [21] observed MX-type precipitates with two kinds of chemical compositions in 9Cr–3W steel with a carbon content from 0.16 to 0.002 mass pct. The

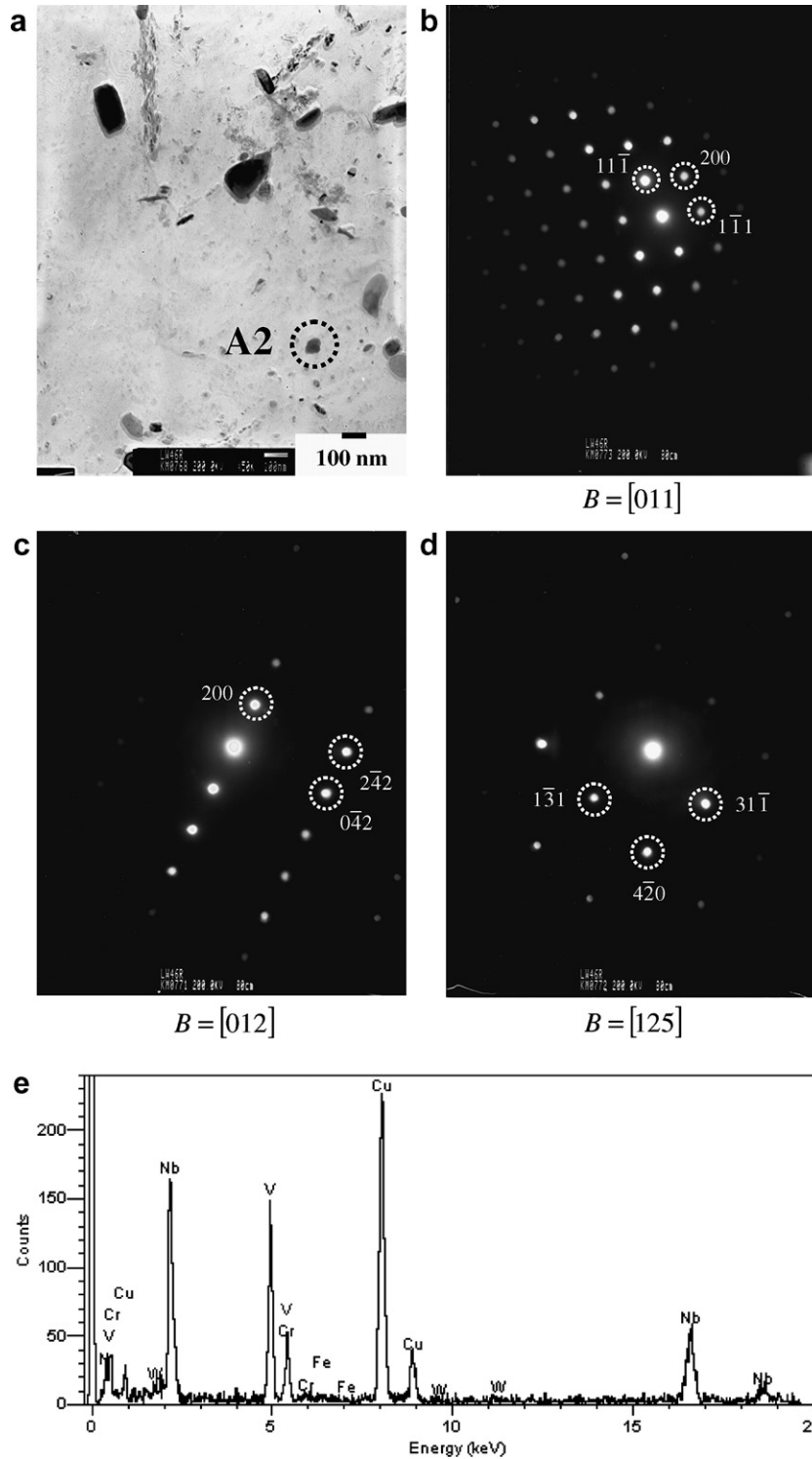


Fig. 3. (a) TEM extraction replica micrograph showing vanadium nitride precipitate A2 marked with circle; (b)–(d) microdiffraction patterns from precipitate A2 in three beam directions of  $[011]$ ,  $[012]$  and  $[125]$ , respectively; (e) EDX spectrum from precipitate A2.

composition of the metal fraction in the MX was about 71V–17Nb–11Cr and 48V–41Nb–11Cr (mass%), respectively, which is strikingly different from the present results (Table 1), i.e., 48V–28Nb–19Cr and 31V–58Nb–8Cr (mass%) corresponding to  $(V_{0.6}Nb_{0.2}Cr_{0.2})N$  and  $(V_{0.45}Nb_{0.45}Cr_{0.1})N$ , respectively.

The observation that two kinds of vanadium nitrides with dramatically different compositions existed in the present steel sample tempered for 2 h at 750 °C gives rise to a question – whether both kinds of vanadium nitrides are stable phases or one of them is just an intermediary phase and will change to another phase with an increasing

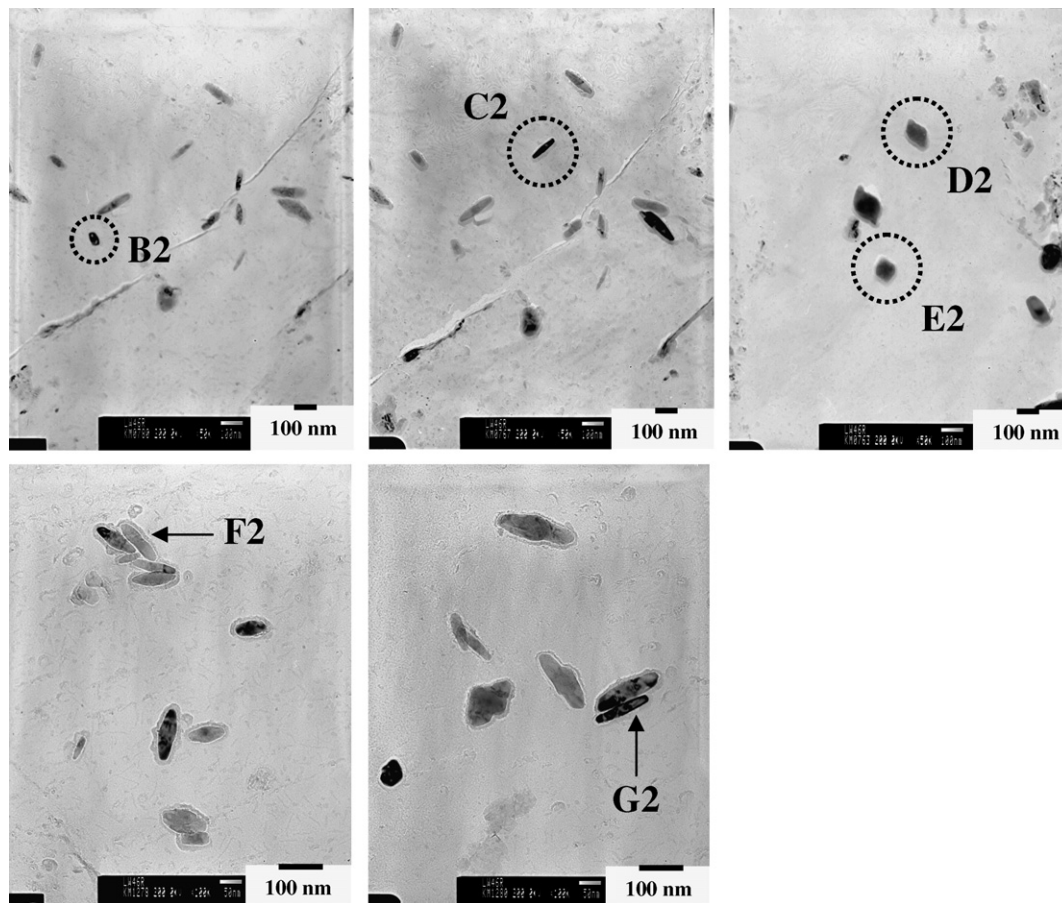


Fig. 4. TEM extraction replica micrographs showing vanadium nitride precipitates B2–G2 marked with circles or arrows.

tempering time. In order to clarify this ambiguity, the steel samples normalized at 1050 °C for 1 h were tempered at 750 °C for 30 min and 5 h, respectively.

Fig. 5 shows the observed vanadium nitrides and their microdiffraction patterns as well as their EDX spectra from the replica of the steel sample tempered for 30 min at 750 °C. Microdiffraction pattern (Fig. 5(b)) from precipitate A3 coincides with that from the vanadium nitride (VN, JCPDS file 25-1252) in the beam direction [111], while the EDX results (Fig. 5(c)) indicates that precipitate A3 has a composition of about 51V–17Cr–15Nb–14N (at.%). Thus, precipitate A3 was determined to be a vanadium-rich MN phase with an approximate expression of  $(V_{0.6}Nb_{0.2}Cr_{0.2})N$ . Meanwhile, from the microdiffraction pattern (Fig. 5(e)), which also coincides with the diffraction pattern of the vanadium nitride (VN, JCPDS file 25-1252) but in the beam direction [112], and the EDX analysis (Fig. 5(f)), precipitate B3 with a composition of about 37V–10Cr–32Nb–14N (at.%) was determined to be a vanadium–niobium-rich MN phase with an approximate expression of  $(V_{0.45}Nb_{0.45}Cr_{0.1})N$ .

The micrographs of the vanadium nitrides taken from the replica of the steel sample tempered for 5 h at 750 °C are shown in Fig. 6(a). Microdiffraction patterns, Fig. 6(b) and (c) obtained from precipitates A4 and B4,

respectively, coincide with those of the vanadium nitride (VN, JCPDS file 25-1252) in the beam directions of [001] and [011]. Combining the EDX spectra, Fig. 6(d) and (e) from precipitates A4 and B4, respectively, precipitates A4 and B4 with a composition of about 52V–15Cr–14Nb–16N (at.%) and 38V–12Cr–37Nb–11N (at.%) were determined to be a vanadium-rich and a vanadium–niobium-rich MN phase, approximately expressed as  $(V_{0.6}Nb_{0.2}Cr_{0.2})N$  and  $(V_{0.45}Nb_{0.45}Cr_{0.1})N$ , respectively.

Since both kinds of vanadium nitrides precipitated in the mean time during the short-term (30 min) tempering stage and there was almost no change in the composition of two vanadium nitrides with an increased tempering time even when tempered for 5 h, it is reasonable to think that the two observed vanadium nitrides,  $(V_{0.6}Nb_{0.2}Cr_{0.2})N$  and  $(V_{0.45}Nb_{0.45}Cr_{0.1})N$ , are probably stable phases.

Table 2 lists some data related to the microdiffraction pattern indexing for the MN-type precipitates A1 and A2. The values of the interplanar spacing  $d$  were calculated by taking an electron wavelength of 0.02508 Å for the accelerating voltage of 200 kV [23] and an effective diffraction camera length of 80 cm. By using  $d$  values and corresponding Miller indices as well as the formulae for  $a$ , the crystal cubic edge for the fcc crystal structure, the mean

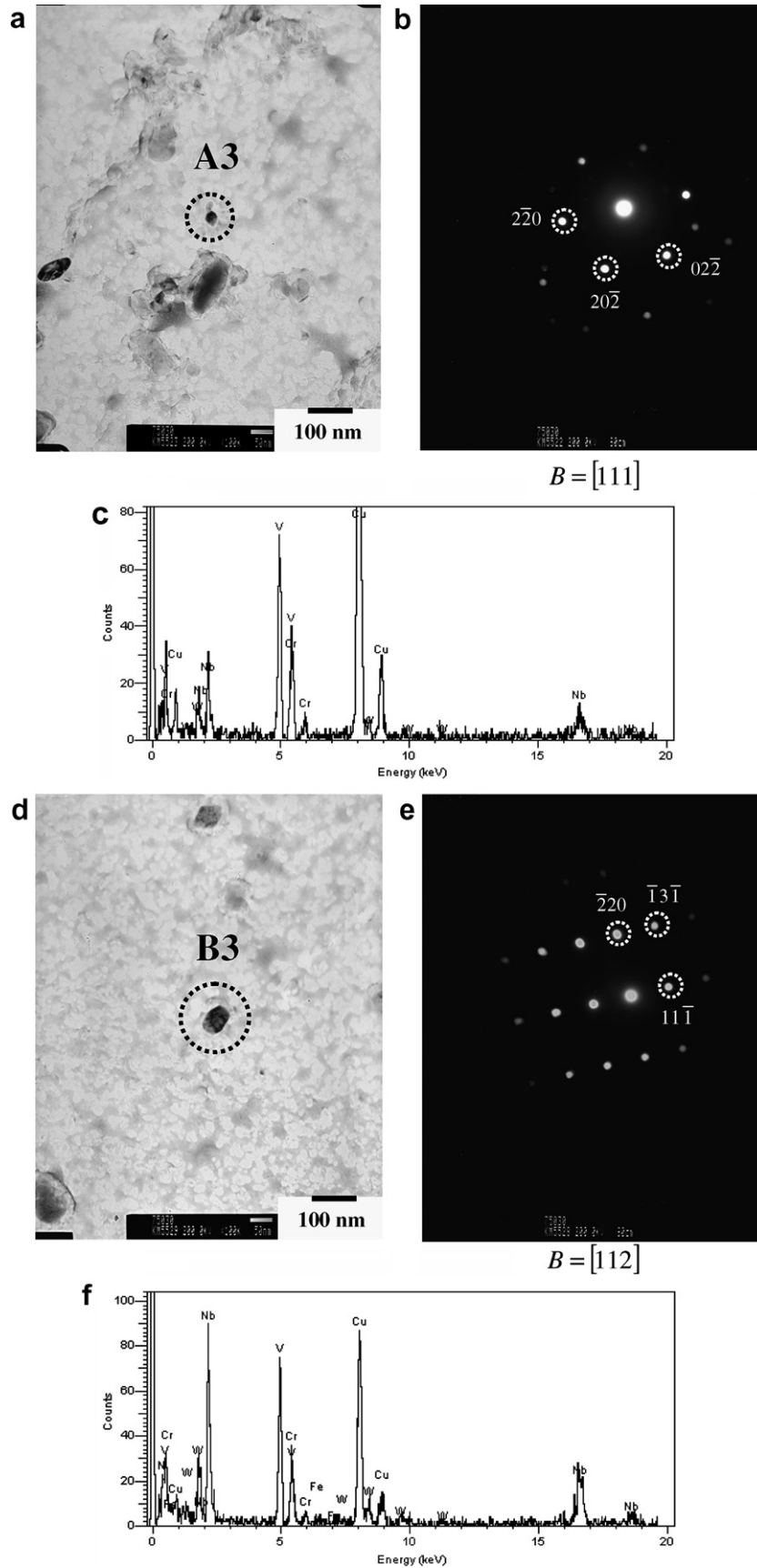


Fig. 5. Vanadium nitride precipitates A3 and B3 in the extraction replica of the steel sample tempered for 30 min at 750 °C. Microdiffraction patterns (b) and (e) with beam directions of [111], [112] and EDX spectra (c) and (f) were from A3 and B3, respectively.

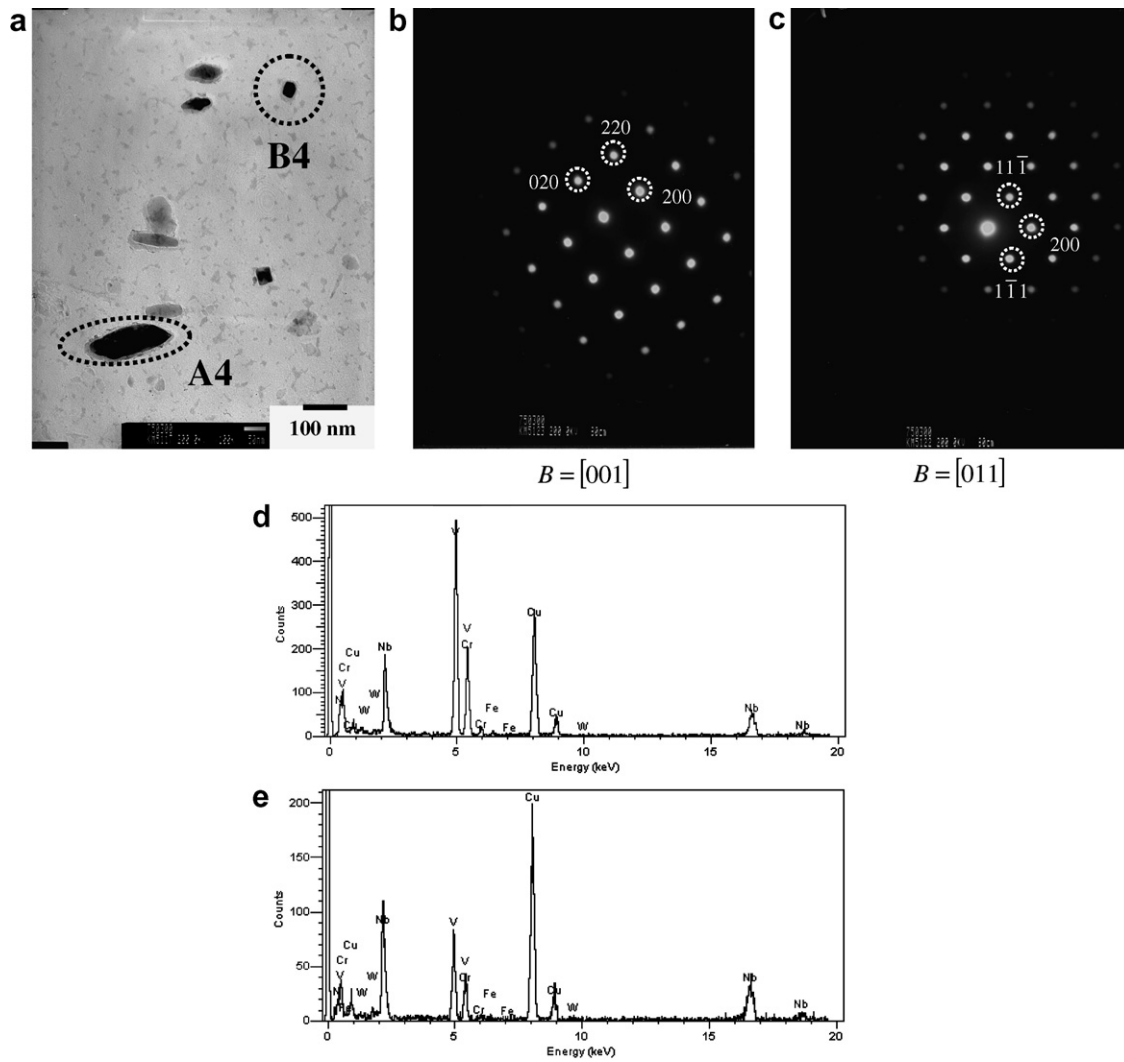


Fig. 6. Vanadium nitride precipitates A4 and B4 in the extraction replica of the steel sample tempered for 5 h at 750 °C. Microdiffraction patterns (b) and (c) with beam directions of [001], [011] and EDX spectra (d) and (e) were from A4 and B4, respectively.

values of the lattice parameter were calculated to be  $\bar{a} = 4.070 \text{ \AA}$  for precipitate phase A1 and  $4.232 \text{ \AA}$  for precipitate phase A2.

Why the lattice parameter calculated from the present microdiffraction patterns is different from that of VN from the JCPDS file (25-1252) is due to the fact that some niobium and chromium is present as a substitution for vanadium thus modifying the lattice parameter of the vanadium–nitride. In order to estimate the change in the lattice parameter due to the niobium and chromium contents in the MN in this work, the relative sizes of the atoms are considered. The lattice parameters of precipitate phases A1 and A2 can roughly be estimated as follows.

Atomic radius of niobium and tungsten atoms is 9.16% and 4.58% larger than that of the vanadium atom, whereas the chromium and iron atoms are 4.58% and 5.34% smaller than the vanadium, respectively. The incorporation of large or small atoms into the lattice of VN will increase or reduce its lattice parameter. The chemical formula of

precipitate phases A1 and A2 can be expressed as  $(V_{0.586}\text{Nb}_{0.1}\text{Cr}_{0.2732}\text{Fe}_{0.0258}\text{W}_{0.015})\text{N}$  and  $(V_{0.4295}\text{Nb}_{0.4653}\text{Cr}_{0.0949}\text{Fe}_{0.0032}\text{W}_{0.0072})\text{N}$ , respectively. The lattice parameter of VN from the JCPDS file 25-1252 is  $4.09 \text{ \AA}$ . Therefore, the lattice parameter can be estimated to approximately be  $a = 4.09 \times (1 + 0.0916 \times 0.1 - 0.0458 \times 0.27 - 0.0534 \times 0.02 + 0.0458 \times 0.01) = 4.0744 \text{ (\AA)}$  for precipitate phase A1 and  $a = 4.09 \times (1 + 0.0916 \times 0.46 - 0.0458 \times 0.09) = 4.2454 \text{ (\AA)}$  for precipitate phase A2. These values agree well with the present experimental data (Table 2).

Also, from Table 1 the chemical formulas of precipitate phases  $(V_{0.6}\text{Nb}_{0.2}\text{Cr}_{0.2})\text{N}$  and  $(V_{0.45}\text{Nb}_{0.45}\text{Cr}_{0.1})\text{N}$  regarding the average chemical compositions can be obtained as  $(V_{0.564}\text{Nb}_{0.1902}\text{Cr}_{0.2138}\text{Fe}_{0.0137}\text{W}_{0.0129})\text{N}$  and  $(V_{0.4284}\text{Nb}_{0.447}\text{Cr}_{0.1082}\text{W}_{0.00109})\text{N}$ , respectively, then their lattice parameters can be estimated as  $\bar{a} = 4.09 \times (1 + 0.0916 \times 0.19 - 0.0458 \times 0.21 - 0.0534 \times 0.01 + 0.0458 \times 0.01) = 4.1187 \text{ (\AA)}$  and  $\bar{a} = 4.09 \times (1 + 0.0916 \times 0.44 - 0.0458 \times 0.1 + 0.0458 \times 0.01) = 4.2380 \text{ (\AA)}$ , respectively.



Table 2

Indexing data for the microdiffraction patterns from the vanadium nitride precipitate phases in the 9% chromium steel and a comparison of the lattice parameters between the experimental data and data from the JCPDS file

<i>R</i> (mm)	$d_{hkl}$ (Å)	<i>hkl</i>	Lattice parameter ( <i>a</i> ) (Å)		Precipitate phase
			Experimental	Estimated and reported	
8.36	2.400	11 $\bar{1}$	4.157	Average: $\bar{a}$ = 4.070 Estimated: $a$ = 4.074; reported: $a$ = 4.09 (JCPDS 25-1252, VN, fcc)	A1 fcc ( $V_{0.6}Nb_{0.2}Cr_{0.2}N$ ) $\bar{a}$ = 4.119 Å (estimated from the average composition given in Table 1)
9.64	2.081	200	4.162		
9.82	2.043	200	4.086		
10.14	1.978	0 $\bar{2}0$	3.957		
13.96	1.427	2 $\bar{2}0$	4.064		
13.98	1.435	2 $\bar{2}0$	4.059		
16.36	1.226	1 $\bar{3}\bar{1}$	4.066		
16.42	1.222	$\bar{1}3\bar{1}$	4.053		
16.51	1.215	3 $\bar{1}\bar{1}$	4.030		
8.13	2.467	1 $\bar{1}\bar{1}$	4.275		
8.41	2.385	11 $\bar{1}$	4.131		
9.40	2.134	200	4.268		
9.58	2.093	200	4.186		
15.47	1.297	$\bar{1}3\bar{1}$	4.302		
15.57	1.288	3 $\bar{1}\bar{1}$	4.273		
21.23	0.9449	0 $\bar{4}2$	4.226		
23.02	0.8714	2 $\bar{4}2$	4.269		
23.64	0.8486	2 $\bar{4}2$	4.157		

*R*: distance of diffraction spots from origin.

$d_{hkl}$ : separation of the (*hkl*) planes.

*hkl*: miller indices.

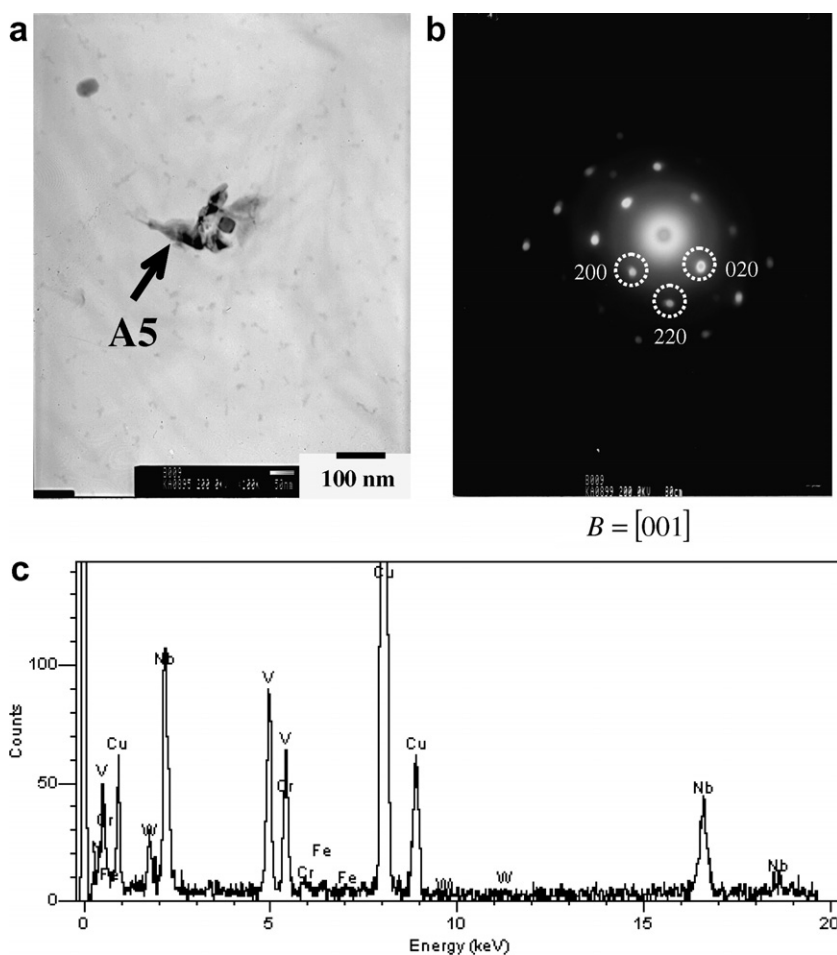


Fig. 7. (a) TEM extraction replica micrograph showing vanadium nitride precipitate A5 in the steel sample normalized at 1100 °C for 1 h; (b) microdiffraction pattern from precipitate A5 in the beam direction of [001]; (c) EDX spectrum from precipitate A5.

The study on the precipitation behavior of 9Cr1MoVNb steel with a carbon content of 0.09 mass pct indicated that the MX precipitates observed in the as tempered condition were carbonitrides [24]. Taneike et al. [21] suggested that one of the observed MX-type carbonitrides in 9Cr–3W steel, 71V–17Nb–11Cr (mass%), was vanadium nitride and another, 48V–41Nb–11Cr (mass%), was mostly regarded as a nitride. Assuming that in the present case precipitates A1 and A2 are vanadium carbides (VC), but not vanadium nitrides (VN), by taking the lattice parameter  $a = 4.30 \text{ \AA}$  of VC (JCPDS file 02-1063), the lattice parameter can be estimated to approximately be  $a = 4.2836 \text{ \AA}$  for precipitate phase A1 and  $a = 4.4635 \text{ \AA}$  for precipitate phase A2, which reveals a larger difference when compared with the experimental data (Table 2) indicating that the two precipitates A1 and A2 do not belong to the VC phase, but belong to the VN phase.

The TEM observation by Taneike et al. [21] indicated that no precipitate except for inconsiderable impurities was present in the 9Cr–3W steels after a normalization at 1100 °C for 30 min, thereby, all the precipitates including MX precipitated during a tempering. In the case of the present steel, however, MN precipitates with a composition of about  $(V_{0.45}Nb_{0.45}Cr_{0.1})N$  were observed in the replica sample of the steel normalized at 1100 °C for 1 h, as shown in Fig. 7. Therefore, in the present 9%Cr steel sample with a tempering condition, the observed  $(V_{0.45}Nb_{0.45}Cr_{0.1})N$  precipitates consist of undissolved particles that exist at the normalizing temperature and particles newly precipitated at the tempering stage, whilst, the observed  $(V_{0.6}Nb_{0.2}Cr_{0.2})N$  precipitates were formed during the tempering.

In view of the present results, it could be expected that the newly observed fine vanadium nitride precipitates must have played an important role in enhancing the creep properties and resistance to irradiation damage for the 9% chromium steel, which should be verified by further experimental work.

#### 4. Conclusions

This work has shown that two kinds of vanadium nitride precipitates with the same fcc crystal structure and different chemical composition coexisted in a 9% chromium steel with a tempering condition. One was a vanadium-rich nitride with a chemical formula of  $(V_{0.6}Nb_{0.2}Cr_{0.2})N$ , a lattice parameter  $\bar{a} = 4.070 \text{ \AA}$ , a plate-like morphology and a size of 80–150 nm in length of the major axis of the precipitate, while the second was a vanadium–niobium-rich nitride with a chemical formula of  $(V_{0.45}Nb_{0.45}Cr_{0.1})N$ , lattice parameter  $\bar{a} = 4.232 \text{ \AA}$ , equiaxial, plate-like and rhombus morphologies, and a size of about 50 nm in diameter or 120–150 nm in length of the major axis of the precipitate. Lattice parameters estimated for the two kinds of precipitates regarding their incorporation of Nb, Cr, Fe and W

into the lattice of VN agreed well with the data from the present microdiffraction patterns, indicating that the precipitates belong to the VN, but do not belong to the VC phase. Observed  $(V_{0.45}Nb_{0.45}Cr_{0.1})N$  precipitates consisted of undissolved particles remaining after a normalizing and the particles newly precipitated during a tempering, whilst,  $(V_{0.6}Nb_{0.2}Cr_{0.2})N$  precipitates originated during the tempering. The two vanadium nitrides seem to be a stable phase, and not an intermediary phase.

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