Cr₂Nb-BASED ALLOY DEVELOPMENT

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ABSTRACT

This paper summarizes recent progress in developing Cr₂Nb/Cr(Nb) alloys for structural use in advanced fossil energy conversion systems. Alloy additions were added to control the microstructure and mechanical properties. Two beneficial elements have been identified among all alloying additions added to the alloys. One element is effective in refining the coarse eutectic structure and thus substantially improves the compressive strength and ductility of the alloys. The other element enhances oxidation resistance without sacrificing the ductility. The tensile properties are sensitive to cast defects, which can not be effectively reduced by HIPping at 1450-1580°C and/or directionally solidifying via a floating zone remelting method.

INTRODUCTION

The objective of this task is to develop new-generation structural materials based on intermetallic alloys for use as critical hot components in advanced fossil energy conversion systems. The intermetallic phase, Cr₂Nb, with a C-15 complex cubic structure,¹ has been selected for this development effort because of its high-melting point (1770°C),²⁻³ relatively low density (7.7 g/cm³),⁴ and potential resistance to oxidation and corrosion.² This intermetallic phase, like many other Laves phases, has a wide range of compositional homogeneity,³ suggesting the possibility of improving its mechanical and metallurgical properties by alloy additions.

The major concern with Cr₂Nb and other A₂B Laves phases is their poor toughness and fracture resistance at ambient temperatures.^{2,5,6} The single-phase Cr₂Nb is very hard (~800 DPH) and brittle at room temperature. Because of the brittleness, our development effort has been concentrated on two-phase structures containing the intermetallic phase Cr₂Nb (hard phase) and the Cr-rich solid-solution phase (ductile phase). Previous studies indicated that the two-phase alloys exhibited plastic deformation under compression tests at

room temperature, with strength much superior to nickel-base superalloys at and above 1000°C.^{5,7,8} Bhandarker^{9,10} has demonstrated that a dispersion of Fe₂Ta Laves-phase particles dispersed within grains of a ferritic steel produced good elevated-temperature strength without causing low-temperature embrittlement. Recently, considerable effort has been devoted to the development of new superalloys containing Laves phases for high-temperature structural use.^{5-8, 10-16}

Our results obtained so far indicate that the two-phase Cr₂Nb/Cr(Nb) alloys have excellent strength for structural use at ultrahigh temperatures (e.g., 1000-1200°C).⁵⁻⁸ Potential applications include hot components in advanced energy conversion systems, advanced heat engines, and high-temperature cutting and grinding tools. Current studies are focused on enhancement of fracture resistance and oxidation resistance. This report summarizes our recent progress on controlling microstructure and improving mechanical properties of Cr₂Nb/Cr(Nb) alloys through alloying additions and material processing. A detailed study of the corrosion behavior of these alloys has been reported separately by Tortorelli et al.¹⁷⁻¹⁹

ALLOY PREPARATION AND PROCESSING

Cr₂Nb/Cr(Nb) alloys weighing 300 g were prepared by arc melting and drop casting in copper molds (1" diam. x 3" long). In order to minimize the interstitial content (e.g., oxygen) in these alloys, high-purity niobium and chromium metal chips were used as charge materials, and the alloys were melted in a high vacuum (10⁻⁵ Pa) furnace. Cast molds were pre-heated to 100-300°C in order to control solidification and to reduce thermal shock during drop casting.

Oxide slags in cracked pieces were observed on the top of alloy buttons and ingots. Sometimes, these oxides were found to be trapped inside alloy ingots. Careful x-ray fluorescence studies indicate that these oxide slags contain mainly zirconium and aluminum. Removal of zirconium and aluminum from the alloys dramatically reduces the slag amount. The drop-cast behavior of the alloys is also sensitive to alloy additions. The drop castability can be substantially improved by certain alloying elements; however, these elements can only be identified by melting and casting practice.

The cast alloy ingots generally contain cast cavities, which vary in size from a couple of microns to as big as several hundred microns. The existing phase diagram of the Cr-Nb system indicates an eutectic temperature of 1620°C for the Cr₂Nb and Cr-rich phases.³ Accordingly, the Cr₂Nb/Cr(Nb) alloys were HIPped at 1450 and 1580°C at a maximum stress of 207 MPa (30 ksi) in order to reduce or eliminate the cavities. Metallographic examination of the ingot interior indicates only marginal reduction in cast

porosities by these HIPpings. This observation indicates extremely high strength at these temperatures and/or a higher melting point of these alloys. Thus, we re-examined the incipient melting point of the chromium niobium (CN) alloys. In this study, alloy specimens were rapidly heated from 1200°C to a preset temperature and kept at that temperature for 6 min., followed by turning off the power. Our studies indicate that the melting point of CN alloys is $\sim 1670 \pm 20^\circ\text{C}$, which is about 50°C above the reported eutectic temperature for the Cr-Nb system. Based on the determined melting point, we plan to HIP the alloys again at a temperature close to 1650°C.

Fig. 1 CN-65 alloy ingot prepared by levitation-zone remelting.

Two alloys, CN-67 and -65, were processed by floating-zone remelting at a velocity of 1.9 - 2.4 cm/hr in high-purity inert gas at the University of Tennessee, using drop-cast ingots as a starting material. Figure 1 shows the rough ingot surfaces, which resulted from evaporation of chromium and formation of surface oxides during crystal growth. Metallographic examination of sectioned ingots shows large holes formed near the ingot surfaces (probably due to chromium evaporation) and cast micro-cavities away from the surface region. This observation indicates that cast cavities in these alloys cannot be completely eliminated by directional solidification through a floating-zone remelting method.

EFFECT OF ALLOYING ADDITIONS ON MICROSTRUCTURE AND PHASE COMPOSITION

During this year, a total of 25 new alloys were prepared. A partial list of the alloy compositions is shown in Table 1. The alloys with 5.6 to 12 at. % Nb basically contain primary Cr-rich solid solution surrounded by the eutectic structure (Fig. 2a), a mixture of Cr_2Nb -type and the Cr-rich phases. The supersaturated Cr-rich solid solution (bright-contrast phase) precipitates out Cr_2Nb -type particles when annealed above 1000°C . The brittle, coarse Cr_2Nb particles in the eutectic structure are, in most cases, interconnected into a skeleton (dark-contrast phase in Fig. 2a) which is believed to be one of the major causes of poor fracture resistance of the CN alloys.

Fig. 2 Optical micrographs of (a) CN-61 and (b) CN-60 annealed for 3 d at 1100°C . (Scale marker is $10\ \mu\text{m}$).

Alloying additions were added to refine the coarse Cr_2Nb eutectic structure in the CN alloys. Among all elements added to the alloys, the element referred to as XM is most effective in refining the interconnected eutectic structure (Fig. 2b). As shown in Fig. 3, the eutectic skeleton is broken into blocky Cr_2Nb -type particles in the alloy containing 6% XM. Another element, referred to as XO, is found to be most effective to date in improving oxidation resistance of the CN alloys. Because of their beneficial effects, a series of alloys containing these two elements were prepared as shown in Table 1. Our

studies of the mechanical properties and oxidation resistance¹⁹ suggest the optimum amount of XM and XO in the same alloy to be 6 and 4%, respectively.

Fig. 3 Backscattered electron images of (a) CN-61 and (b) CN-60 annealed for 3 d at 1100°C. (Scale marker is 10 μ m).

Table 1. Nominal Alloy Compositions of Selected CN Alloys

Alloy Number	Composition (at. %)
CN-60	Cr-12.0Nb-4.0Re-2.0Al-6.0XM
CN-80	Cr-12.0Nb-1.5Al-6.0XM
CN-72	Cr-12.0Nb-1.5Al-0.1Zr-6.0XM
CN-61	Cr-12.0Nb-4.0Re-2.0Al-4.0XO
CN-81	Cr-10.0Nb-1.5Al-6.0XM-4.0XO
CN-67	Cr-8.0Nb-1.5Al-0.1Zr-6.0XM
CN-64	Cr-5.6Nb-1.5Al-0.1Zr-4.0XM
CN-65	Cr-5.6Nb-1.5Al-0.1Zr-6.0XM
CN-69	Cr-5.6Nb-1.5Al-6.0XM
CN-70	Cr-5.6Nb-1.5Al-0.1B-6.0XM
CN-68	Cr-5.6Nb-1.5Al-0.1Zr-4.0Re-6.0XM
CN-66	Cr-5.6Nb-1.5Al-0.1Zr-10.0XM
CN-71	Cr-5.6Nb-1.5Al-0.1Zr-15.0XM
CN-74	Cr-5.6Nb-1.5Al-0.1Zr-6.0XM-4.0XO
CN-75	Cr-5.6Nb-1.5Al-0.1Zr-6.0XM-6.0XO
CN-76	Cr-5.6Nb-1.5Al-0.1Zr-6.0XM-8.0XO

Figure 4 shows the effect of heat treatment on the microstructures of the CN-65 alloy containing 5.6% Nb. Fine precipitates distribute uniformly across grains and some coarse Cr_2Nb particles are observed at grain boundaries in the specimen (Fig. 4a) annealed at 1100°C for 3 days. HIPping at 1450°C apparently dissolved a major part of the fine precipitates within grains but did not remove the coarse Cr_2Nb particles at grain boundaries (Fig. 4b). A few cavities are visible in the HIPped specimen: Figure 5 shows the microstructure of the alloy containing 6% XM and 6% XO, where the eutectic structure can be easily identified. Comparison of Fig. 5 with Figs. 3b and 4a suggests that XO additions stabilize the classic eutectic structure.

The composition of phases in CN alloys was determined by electron microprobe analyses. Table 2 summarizes the composition of the phases determined by wavelength-dispersive spectrometry (WDS). The Cr-rich solid solution, the matrix phase, contains a low level of niobium, $\sim 1\%$. The Cr_2Nb -type phase, on the other hand, contains more than 20% Nb, and the exact amount of niobium in the Cr_2Nb -type phase depends on the partitioning of alloying additions in the CN alloys. The element XM partitions more or less equally in the Cr_2Nb and the Cr-rich phases. The analysis of the composition of Cr_2Nb -type phase in CN-65 indicates that XM essentially occupies the niobium sites. The element XO in CN-75 partitions strongly in the Cr_2Nb -type phase, as indicated in Table 2. The partitioning ratio of XO in the Cr_2Nb -type and Cr-rich phases is 5.6 to 1. Unlike XM,

Fig. 4 Optical micrographs of CN-65 (a) annealed for 3 d at 1100°C , and (b) HIPped for 2 h at 1450°C . (Scale marker is $10\ \mu\text{m}$).

Fig. 5 Backscattered electron image of CN-75 annealed for 3 d at 1200°C. (Scale marker is 10 μ m).

Table 2. WDS Compositional Analysis of CN Alloys*

Alloy Number	Cr ₂ Nb-type Phase (at.%)	Cr-rich Matrix Without Fine Cr ₂ Nb Precipitates (at.%)	Cr-rich Matrix With Fine Cr ₂ Nb Precipitates (at.%)
CN-65	Cr = 67.0	Cr = 90.9	Cr = 88.2
	Nb = 25.8	Nb = 1.2	Nb = 4.4
	XM = 5.4	XM = 6.7	XM = 6.2
	Al = 1.8	Al = 1.2	Al = 1.2
	Zr = 0.0	Zr = 0.0	Zr = 0.0
CN-60	Cr = 63.4		Cr = 79.0
	Nb = 25.4		Nb = 7.6
	XM = 5.0		XM = 6.8
	Re = 4.2		Re = 5.1
	Al = 2.0		Al = 1.5
CN-75	Cr = 55.0	Cr = 90.2	Cr = 86.8
	Nb = 22.3	Nb = 0.6	Nb = 3.0
	XM = 7.9	XM = 5.2	XM = 5.5
	XO = 14.0	XO = 2.5	XO = 3.4
	Al = 0.8	Al = 1.5	Al = 1.3
	Zr = 0.0	Zr = 0.0	Zr = 0.0

*Specimens annealed for 3 d at 1100°C or 1200°C.

XO mainly occupies the Cr sites instead of the niobium sites. The elements rhenium and aluminum partition approximately equally in the two phases. Zirconium was not detected in the CN alloys, and was probably lost due to its formation of ZrO₂ slags during melting.

The alloys CN-60 and CN-61 were examined by transmission electron microscopy (TEM). Both alloy specimens were annealed for 3 d at 1100°C, and then cut into 3 mm disks and ground to a thickness of approximately 50 μm , followed by ion milling at 6 kV. The specimens were examined in a Philips CM30 at 300 kV. Energy dispersive spectroscopy (EDS) was performed with an EDAX 9100 spectrometer. The EDS analyses were made on precipitates that intersected the ion milling perforations, thereby minimizing the contributions from surrounding matrix material.

Both alloys showed a mixture of the Cr-rich solid solution and Cr_2Nb -type phases. The small precipitates in the Cr-rich matrix ranged up to 0.4 μm in diameter. Figure 6a shows the eutectic structure (dark areas) in CN-61 (containing 4% XO) with only a few secondary precipitates visible. Figure 6b shows a region in CN-60 (containing 6% XM) with an extensive secondary precipitation. EDS analyses of the matrix versus the precipitates were made along the edge of the specimen as shown in Fig. 6b. Figure 6c shows dislocations in the Cr-rich matrix in CN-60. While many of the dislocations appeared to have interacted with small precipitates by bowing between them, many other dislocations have formed low-angle network structures.

EDS results were averaged and are shown in Table 3. Basically, the data in Table 3 agree well with those in Table 2 determined by electron microscopic analysis. In CN-60, the XM and rhenium additions did not preferentially partition to Cr_2Nb particles. Only about 1% of niobium remained in solution, with the rest precipitating out. In CN-61, the XO additions did preferentially precipitate out with the niobium in Cr_2Nb -type particles, while again the rhenium levels were similar in both matrix and in the Cr_2Nb -type phase. Apparently, no zirconium was retained by the alloy.

MECHANICAL PROPERTIES

The cast CN alloys, even in the HIPped conditions, contain cast defects such as cavities. Since the compressive properties are much less sensitive to these defects (as compared with tensile properties), the mechanical properties of the CN alloys were determined mainly by compressive tests.

Compressive Properties

The compressive properties of the CN alloys were determined at temperatures to 1200°C. Alloy specimens were annealed for 3 d at 1100°C prior to testing at room

temperature in air and at elevated temperatures in vacuum. Tables 4 and 5 summarize the results at different temperatures. For the alloys containing 12 at.% Nb, the yield strength

Fig. 6 TEM micrographs of (a) the eutectic Cr_2Nb structure in CN-61, (b) block particles in CN-60, and (c) dislocations in CN-60. All specimens were annealed for 3 d at 1100°C.

Table 3. EDS/TEM Compositional Analysis of CN-60 and CN-61

Alloy No.	Concentrations (at.%)					
	Cr	Nb	Re	Al	XM	XO
CN-60						
nominal	76	12	4	2	6	
matrix	86	1	5	0	7	
secondary precipitation	52	39	5	0	3	
large precipitates	63	28	4	0	5	
CN-61						
nominal	78	12	4	2		4
matrix	91	1	3	1		5
large precipitates	53	28	4	1		13
secondary precipitation	57	29	5	0		15

Table 4. Compressive Properties of CN Alloys Tested at Room Temperature in Air

Alloy No.	Concentration of Key Elements (at.%)	Strength, MPa (ksi)		Ductility (%)
		Yield	Ultimate	
CN-4	12Nb	960 (139)	1760 (255)	5.4
CN-44	12Nb-4Re	1230 (179)	2050 (298)	4.3
CN-60	12Nb-4Re-6XM	1793 (260)	2546 (369)	8.5
CN-61	12Nb-4Re-4XO	1395 (202)	1946 (282)	5.2
CN-7	6Nb	702 (102)	1261 (183)	9.5
CN-65	5.6Nb-6XM	1116 (161)	2228 (323)	16.0
CN-65*	5.6Nb-6XM	1097 (159)	2116 (307)	18.5
CN-68	5.6Nb-4Re-6XM	1111 (161)	2023 (293)	17.7
CN-70*	5.6Nb-0.1B-6XM	1047 (152)	2059 (297)	16.3
CN-75	5.6Nb-6XM-6XO	1300 (189)	2080 (302)	16.2

*HIPped at 1450°C and annealed 3 d/1100°C.

at room temperature increases significantly by alloying with XM, rhenium, and XO.

However, only XM improves the room-temperature ductility. XM is also very effective in improving both the strength and ductility of the CN alloys containing 5.6-6% Nb at room

temperature (Table 4). The alloy CN-65 containing 5.6% Nb and 6% XM showed a yield strength of 1097 MPa and a ductility of 18.5% at room temperature. Alloying with 6% XO together with 6% XM appears not to lower the room-temperature ductility of the 5.6% Nb alloy.

At 1000 and 1200°C, both XM and rhenium effectively increase the compressive strength of the CN alloys containing 5.6 and 12% Nb (Table 5). The ductility of the alloys is not sensitive to alloying additions, and all the alloys showed ductility of more than 23%. HIPping at 1450°C appears not to affect the ductility at either room or elevated temperatures. The CN alloys are much stronger than Ni-base superalloys, whose strength drops to about zero at 1200°C.

Table 5. Compressive Properties of CN Alloys Tested at 1000 and 1200°C in Vacuum

Alloy No.	Concentration of Key Elements - (at.%)	Strength, MPa (ksi)		Ductility (%)
		Yield	Ultimate	
1000°C				
CN-4	12Nb	685 (99)	856 (124)	22.8
CN-44	12Nb-4Re	855 (124)	1061 (154)	26.2
CN-60	12Nb-4Re-6XM	896 (130)	1632 (237)	29.2
CN-61	12Nb-4Re-4XO	741 (107)	1257 (182)	28.5
CN-7	6.0Nb	436 (63)	738 (107)	32.7
CN-65	5.6Nb-6XM	634 (92)	1203 (174)	33.8
CN-68	5.6Nb-4Re-6XM	675 (98)	1118 (162)	26.5
CN-70*	5.6Nb-0.1B-6XM	778 (113)	1394 (202)	23.1
CN-75	5.6Nb-6XM-6XO	710 (103)	1302 (189)	32.4
1200°C				
CN-4		363 (53)	487 (71)	>31.5
CN-44		432 (63)	566 (82)	> 4.1
CN-60		535 (78)	664 (96)	> 5.6
CN-7		258 (37)	309 (45)	31.4
CN-65		404 (59)	547 (79)	30.4

*HIPped at 1450°C.

Tensile Properties

Tensile properties of HIPped and/or directionally solidified specimens were determined at room temperature and 1000°C. As mentioned before, these specimens contain cast defects (even after HIPping), and only the highest fracture strength for each alloy are reported in Table 6. The alloys showed a fracture strength at a level of 200-500 MPa, which appears to be independent of alloy composition. There is no apparent difference in fracture strength at room temperature and 1000°C, suggesting that the fracture process is dominated by cast defects in the alloys. Further studies are required to reduce cast defects by either HIPping at higher temperature (>1580°C) or hot extruding the CN alloys. A recent study of two-phase alloys based on Nb and Nb₅Si₃ indicates that their tensile ductility and strength can be dramatically improved by hot extrusion of the alloys at 1426°C.

Table 6. Tensile Properties of CN Alloys Tested
at Room Temperature in Air and 1000°C in Vacuum

Alloy No.	Alloy Composition (at.%)	Treatment Condition*	Fracture Stress, MPa (ksi)
Room Temperature			
CN-70	Cr-5.6Nb-1.5Al-0.1B-6XM	HIP/1450°C	326 (47.3)
CN-65	Cr-5.6Nb-1.5Al-0.1Zr-6XM	HIP/1580°C	266 (38.6)
CN-65	Cr-5.6Nb-1.5Al-0.1Zr-6XM	DS	367 (53.3)
CN-67	Cr-8Nb-1.5Al-0.1Zr-6XM	DS/HIP/1580°C	207 (30.1)
CN-74	Cr-5.6Nb-1.5Al-0.1Zr-6XM-4XO	HIP/1580°C	416 (60.4)
1000°C			
CN-70	Cr-5.6Nb-1.5Al-0.1B-6XM	HIP/1450°C	478 (69.4)
CN-65	Cr-5.6Nb-1.5Al-0.1Zr-6XM	HIP/1580°C	360 (52.2)
CN-67	Cr-8Nb-1.5Al-0.1Zr-6XM	DS/HIP/1580°C	382 (55.5)
CN-74	Cr-5.6Nb-1.5Al-0.1Zr-6XM-4XO	HIP/1580°C	248 (36.0)

HIP = hot isostatically pressing

DS = directional solidification by levitation zone remelting

Fracture surfaces of the tensile specimens tested at room temperature and 1000°C were examined using a scanning electron microscope operated at 10 kV. The CN alloys showed mainly cleavage fracture at both temperatures with a more ductile appearance at 1000°C. Cast defects are visible on the fracture surfaces.

Creep Resistance

We recently initiated creep tests of CN alloys containing XM. The alloy CN-70 showed no measurable creep deformation at 1200°C at a stress of 55 MPa (8 ksi), indicating excellent creep resistance. The alloy exhibited a creep rate of $1.5 \times 10^{-7} \text{ h}^{-1}$ at 1000°C at a stress of 345 MPa (50 ksi). The alloy is much more creep resistant than Ni-base superalloys.

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