FABRICATION OF 500-kg HEAT OF V–4Cr–4Ti* H. M. Chung, H.–C. Tsai, and D. L. Smith (Argonne National Laboratory) and R. Peterson, C. Curtis, C. Wojcik, and R. Kinney (Teledyne Wah Chang Albany)

OBJECTIVE

On the basis of excellent properties that were determined for a laboratory–scale heat, V–4Cr–4Ti has been identified previously as the primary vanadium–based candidate alloy for application in fusion reactor structural components. The objective of this work is to produce a large–scale (500–kg) ingot and fabricate various plates and sheets from the ingot, thereby demonstrating a reliable method of fabricating an industrial–scale heat of V–4Cr–4Ti that exhibits excellent properties.

SUMMARY

A 500-kg heat of V-4Cr-4Ti, an alloy identified previously as the primary vanadium–based candidate alloy for application in fusion reactor structural components, has been produced. The ingot was produced by electron–beam melting using screened high–quality raw materials of vanadium and titanium. Several ≈63.5–mm–thick bars were extruded from the ingot, and plates and sheets of various thicknesses ranging from 0.51 to 12.7 mm were fabricated successfully from the extruded bars. The chemical composition of the ingot and the secondary fabrication procedures, specified on the basis of experience and knowledge gained from fabrication, testing, and microstructural examination of a laboratory–scale heat, were found to be satisfactory. Charpy–impact tests showed that mechanical properties of the large–scale heat are as good as those of the laboratory–scale heat. This demonstrates a method of reliable fabrication of industrial–scale heats of V–4Cr–4Ti that exhibit excellent properties.

INTRODUCTION

Vanadium-base alloys have significant advantages over other candidate alloys (such as austenitic and ferritic steels) for use as structural materials in fusion devices. In the past, extensive investigations have been conducted on the swelling behavior, tensile properties, impact toughness, and microstructural evolution of V, V-Ti, V-Cr, V-Cr-Ti, and V-Ti-Si alloys after irradiation by fast neutrons at 420°C–600°C. These investigations revealed that V-Cr-Ti alloys containing ≈4 wt.% Cr, ≈4 wt.% Ti, 400-1000 wt. ppm Si, and <1000 wt. ppm O+N+C were most desirable because they exhibit superior physical and mechanical properties.[1–6] These results were obtained, however, on small (<30–kg) laboratory–scale heats, including a small heat (ANL ID BL–47) of V-4Cr-4Ti that exhibited excellent resistance to irradiation–induced embrittlement, swelling, and helium embrittlement.[7–9] In the meantime, a relatively larger heat of V–5Cr–5Ti (ANL ID BL–63) and a small heat of V–5Cr–3Ti (ANL ID BL–54), fabricated subsequently under procedures significantly different from those of the small laboratory heat of V–4Cr–4Ti, were found to exhibit less–desirable mechanical properties despite the small differences in alloying element contents.[10] Based on these backgrounds, a joint campaign was initiated at Argonne National Laboratory and Teledyne Wah Chang (Albany, Oregon) to identify an optimal fabrication procedure of the V–(4–5)Cr–(3–5)Ti alloys and to demonstrate of a reliable method of producing an industrial–scale heat of V–4Cr–4Ti that exhibits the excellent properties reported in previous investigations.

SPECIFICATIONS AND PROCEDURES

The production campaign consisted of a large–scale (500–kg) melting of a V–4Cr–4Ti ingot; extruding the ingot into rectangular bars; and subsequent rolling and heat–treatments to manufacture final

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products in the form of plates and sheets of various thickness; analyses of chemical composition and phase structure; and Charpy–impact testing to determine the ductile–brittle transition temperatutre (DBTT). The latter information is described in a separate article in this report.[11]

1. Specification of Chemical Composition

The specification of chemical composition was carefully evaluated to reflect the valuable experience and knowledge gained from fabrication, testing, and microstructural examination of the laboratory–scale heats of of V–Ti and V–Cr–Ti alloys, in particular, V–4Cr–4Ti (BL–47), V–5Cr–3Ti (BL–54), and the relatively larger heat of V–5Cr–5Ti (BL–63).[3,5,10] Particular attention was given to control of the following impurities: Nb and Mo (minimize to ensure low neutron activation); Cu (minimize to suppress Cu–rich vanadium oxycarbochlorides);[10] Si (optimize to 400–1000 wppm to suppress swelling);[5] O, N, and C (limit the combined concentration to <1000 wppm); S, P (minimize to avoid segregation to grain boundary and precipitation);[10] and Cl, Ca, K, Na, and B (minimize to avoid formation of the vanadium–based precipitates oxycarbochlorides, vanadates, and borides).[10] The final specification of ingot chemistry is listed in the following:

1.1 Ingot Chemical Specifications

Element	Composition	Element	Composition
Cr	4.0 ± 0.5 wt.%	Ti	4.0±0.5 wt.%
Si	400 - 1000[a]	O	<400[a]
N	<200	C	<200
Al	<200	Fe	<200
Cu	<10	Mo	<10
Nb	<10	Cl	<2
P	<10	S	<10

[a]Concentrations in wt. ppm.

Use only low–chlorine (<5 wppm) double– or triple–melted titanium feedstock in making the ingot.

1.2 Alloying Component Inhomogeneity

Local inhomogeneity of Cr and Ti shall not exceed ±0.3 wt.%.

2. Screening of Raw Materials

The source of the last group of impurities , except for Ca and B (i.e., Cl, K, and Na), is known to be primarily low–quality Ti, which is normally produced by the Kroll process. Therefore, only double– or triple–melted Ti was used in ingot melting. This is in contrast to the use of sponge titanium in melting the ingots of the V–5Cr–5Ti (BL–63) and V–5Cr–3Ti (BL–54), the two heats reported to exhibit inferior mechanical properties.[10]

Chemical composition of the raw vanadium designated for melting of the ingot was also checked (Table 1). At the same time, a piece of the raw vanadium was set aside and machined into 1/3–size Charpy–impact specimens. Normally, unalloyed vanadium of good quality (e.g., iodide[12] or electrorefined[13] vanadium containing low O, N, Ca, and B) should exhibit a DBTT lower than –200°C. However, unusually higher DBTTs have been observed occasionally in the past, in particular for some of the unalloyed vanadium produced by the calcium–reduction process.[13,14] Specimens from one of the

unalloyed vanadium (BL–51) that were impact–tested in a previous investigation exhibited a DBTT as high as –150°C.[2] From a subsequent microstructural characterization by analytical electron microscopy, the unusually high DBTT of the heat (BL–51) has been attributed to dense precipitation of very fine (<60 nm diameter) calcium vanadates.[10] In view of these observations, impact tests were conducted on the 1/3–size Charpy specimens of the raw vanadium, and it was verified that the DBTT of the raw material is lower than –196°C.

3. Specification of Secondary Fabrication and Conversion

The procedures for extrusion, secondary fabrication, and conversion to plates and sheets were specified as follows to closely reproduce the procedures used in fabricating the excellent laboratory heat of V–4Cr–4Ti (ANL ID BL–47):

1. Extrude ingot in stainless steel jacket at 1150°C into 63–mm–thick slab.
2. Subsequent warm rolling at 400°C to desired final dimensions.
3. No more than 15 and 50% thickness reduction, respectively, per roll and between anneals.
4. Annealing at 1050°C for 1 h in high–vacuum furnace before each rolling.
5. Annealing at 1050°C for 2 h in high–vacuum furnace for all final products (thicker than ≈3 mm).

FINAL PRODUCTS

The ≈500-kg ingot was produced by electron–beam melting for constituent homogeneity. This is in contrast to small laboratory heats (including Heat BL–47 and a new 13–kg heat of V–5Cr–5Ti, BL–72) that were produced by vacuum–arc melting. The results of chemical analyses, averaged over three measurements on 63–mm–thick extruded bars, are given in Table 1. For comparison, the chemical composition of the excellent laboratory heat of V–4Cr–4Ti (ANL ID BL–47) is also given in the table. The machined cast ingot was encased in a stainless jacket and extruded at 1150°C into bar with a nominal thickness of 63.5 mm (2.5 in.). Three pieces of the ≈63.5–mm–thick extruded bars are shown in Fig. 1.

Table 1. Chemical composition (impurities in wppm) of the industrial–scale (500 kg) heat of V–4Cr–4Ti and the V raw stock used to melt the ingot.

Heat ID	Material	Cr	Ti	Cu	Si	O	N	C	S	P	Ca	Cl	B
820630	V raw stock	<100	<50	<50	780	200	62	75	10	<30	–	<2	<5
832665	V–4Cr–4Ti	3.8 wt.%	3.9 wt.%	<50	783	310	85	80	<10	<30	<10	<2	<5

Following extrusion, ≈72 kg (or 17%) of the extruded bar (≈430 kg recovered) has been used to produce plates and sheets of nominal thicknesses of 12.7, 6.35, 3.81, and 1.02 mm. Plates with a thickness of 3.81 mm and greater were produced by multiple-pass cross–rolling at ≈400°C; the thinner, 1.02-mm-thick sheets were produced by further rolling at room temperature. The thickness reduction was limited to <15% per pass. Annealing treatments of the plates were conducted at 1050°C-1070°C for 2 h in a vacuum better than ≈1.33×10^{-3} Pa.

The chemical composition of the heat, similar to that of the excellent laboratory heat (BL–47), conforms with the specification. As expected, results of impact tests of 1/3–sizes Charpy specimens machined from the 3.81–mm plate showed also that mechanical properties of the large–scale heat are as good as those of the laboratory–scale heat.[11]

Fig. 1. Three pieces of ≈63–mm–thick bars extruded from 500–kg industrial–scale heats (ID 832665) of V–4Cr–4Ti.

CONCLUSION

A scale–up (500-kg) heat of V-4Cr-4Ti, an alloy identified previously as the primary vanadium–based candidate alloy for application in fusion reactor structural components, has been produced successfully by electron–beam melting of screened high–quality raw materials of vanadium and titanium. Several ≈63.5–mm–thick bars were extruded from the ingot, and plates and sheets of various thicknesses ranging from 0.51 to 12.7 mm were fabricated successfully from the extruded bar. The chemical composition of the ingot and the secondary fabrication procedures, specified on the basis of experience and knowledge gained from fabrication, testing, and microstructural examination of a laboratory–scale heat, were found to be satisfactory. Results of Charpy–impact tests showed that mechanical properties of the large–scale heat are as good as those of the laboratory–scale heat. This demonstrates a method of reliable fabrication of industrial–scale heats of V–4Cr–4Ti of excellent properties.

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