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# Tensile properties of a series of V-4Ti-4Cr alloys containing small amounts of Si, Al and Y, and the influence of helium implantation

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# Abstract

V–Ti–Cr type alloys containing Si, Al and Y have been developed to improve proof-oxidation properties and high temperature strength. In order to optimize the composition of Si, Al and Y, seven V–Ti–Cr type alloys containing Si, Al and Y up to 0.5 wt% were prepared. Tensile tests were carried out at temperatures from 300 to 1123 K. From the results of the tests, the dependence of yield stress and ultimate tensile strength on Si, Al and Y concentration is low at these temperatures. Total elongation of the alloys tested at 923 and 1123 K increase with increasing concentration of Si, Al and Y. Helium implantation up to about 50 appm by an accelerator was also carried out. The loss of total elongation at 923 and 1123 K depends on the concentration of Si, Al and Y. From the results of helium implantation, it is suggested that the appropriate concentration of Si, Al and Y for V–4Ti–4Cr alloys is between 0.1 and 0.5 wt%, respectively. © 1998 Elsevier Science B.V. All rights reserved.

### 1. Introduction

Vanadium alloys are considered to be candidate structural materials for fusion reactors because of their high-heat loading capability and low activation properties. The alloys of V-(4-5)Ti-(4-5)Cr type were studied as a reference alloy [1], because of their low swelling behavior after irradiation [2] and low DBTT [3]. Vanadium alloys, however, are very susceptible to oxidation. Interstitial impurity atoms such as oxygen affect both irradiation behavior and baseline properties of the alloy [4]. Therefore, it is important to improve their oxidation resistance from the practical point of view. Alloys containing small amounts of Si, Al and Y, in order to improve proof-oxidation properties, have been studied previously [5–10]. This V–Ti–Cr–Si–Al–Y type alloy

when very high amounts of helium were implanted by an accelerator [11]. This may correlate to solution strengthening by alloying elements [11]. It is important to determine the appropriate concentration of Si, Al and Y for V-4Ti-4Cr type alloys. This optimization procedure should consider all material properties including fabrication, baseline properties and irradiation behavior. Tensile tests were performed to study one of the baseline properties of the alloy. Seven variations of Si, Al and Y contents in V-Ti-Cr type alloys were examined at a temperature ranging from ambient temperature to 1123 K. One of the characteristic conditions in fusion applications is a large amount of helium generation by nuclear transmutation in materials by  $(n,\alpha)$  reaction. Therefore, it is one of the important issues for optimization of the alloy composition. In this paper, tensile properties of the alloys and influence of helium implantation are described.

showed a very good combination of irradiation properties such as low swelling behavior and mechanical

properties [8]. Loss of ductility of the alloy was observed

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Table 1					
Chemical	analysis	of	vanadium	alloys	(wt%)

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	Alloy	Heat#	V	Ti	Cr	Si	Al	Y	С	0	Ν	Н
a	V-4Ti-4Cr-0.5Si-0.5Al-0.5Y	KAV9601	Bal.	3.99	3.96	0.46	0.49	0.20	0.0173	0.029	0.013	0.0040
b	V-4Ti-4Cr-0.3Si-0.3Al-0.3Y	KAV9603	Bal.	4.07	3.96	0.34	0.29	0.10	0.0142	0.034	0.013	0.0038
с	V-4Ti-4Cr-0.1Si-0.1Al-0.1Y	KAV9605	Bal.	4.08	3.96	0.14	0.08	0.05	0.0165	0.071	0.013	0.0038
d	V-4Ti-4Cr-0.5Si-0.5Y	KAV9607	Bal.	4.10	4.00	0.46	-	0.22	0.0164	0.026	0.013	0.0034
e	V-4Ti-4Cr-0.1Si-0.1Y	KAV9610	Bal.	4.09	3.92	0.14	-	0.05	0.0146	0.078	0.012	0.0029
f	V-4Ti-4Cr	KAV9611	Bal.	4.04	3.95	-	-	-	0.0224	0.115	0.012	
g	V-5Ti-5Cr-1Si-1Al-1Y	KAV9612	Bal.	5.12	4.92	1.04	1.03	0.72	0.0273	0.026	0.013	0.0040
h	V-5Ti-5Cr-1Si-1Al-1Y	KAV6	Bal.	4.79	4.01	0.85	0.95	0.77	0.0126	0.014	0.005	

# 2. Experimental

Seven alloys of V-Ti-Cr-Si-Al-Y type were used in this study. The chemical analysis of these alloys is shown in Table 1. In the table each alloy is designated by an alphabetical symbol for simplification. The six alloys (a)-(f) are V-4Ti-4Cr type alloys containing a range of 0-0.5 wt% of Si, Al and Y. The alloys of (g) V-5Ti-5Cr-1Si-1Al-1Y were prepared to compare with previous work, which was designated (h). Buttons of alloys were arc-melted. The buttons of about 130 g weight were encapsuled in a box made of stainless steel, and hot pressed to about 5 mm in thickness after annealing at 1273 K for 3.6 ks, followed by removing the surface layers. Consequently the ingots were about 3 mm in thickness. Sheets of 0.25 mm thickness were obtained by cold rolling. The measured concentration of Y is a little lower than the nominal amount. It is considered that Y was scavenging oxygen in the ingots during the arcmelting process, and then the yttrium oxides precipitate on the surface, which were removed by grinding. Therefore, the Y contents was reduced depending on oxygen contents in raw materials. The miniature tensile specimens with a gauge section of 5 mm long and 1.2 mm wide were punched out from the sheets. The specimens were annealed at 1373 K for 3.6 ks in vacuum to obtain a fully recrystallized condition with a mean grain size of about 20 µm. Tensile tests were carried out using an Instron-type machine with a external cantalum heater in a vacuum of  $1 \times 10^{-3}$  Pa at a strain rate of  $6.7 \times 10^{-4}$ s<sup>-1</sup> at temperatures from 300 to 1123 K. After tensile tests, specimens were examined by scanning electron microscopy (SEM) in order to characterize the fracture surface. Helium ion of 36 MeV was implanted into selected alloys by Tohoku University cyclotron accelerator using a tandem-type energy degrader wheels [12]. To obtain uniform helium distribution along the implanted direction, energy degrader wheels consisting of aluminum foils with 525 steps were used. Total amount of helium in the specimen was evaluated to be about 50 appm with displacement damage of about 0.02 dpa. Microstructural observation was carried out for selected specimens using transmission electron microscopy

(TEM). TEM observation also perfored after post-implantation annealing at 1123 K for 3.6 ks in a vacuum of  $1 \times 10^{-3}$  Pa.

#### 3. Results and discussion

#### 3.1. Tensile properties

Fig. 1 shows typical stress-strain curves of the specimens tested at 300, 923 and 1123 K. Results of four alloys out of seven are shown Fig. 1. The alloy designation is same as in Table 1. The tensile curves of all alloys are similar at a testing temperature of 300 K. At 923 K, a serrated flow is observed and total elongation decreased with decreasing concentration of Si, Al and Y. The serrated flow, that is, corresponding to dynamic strain aging, is observed in a narrower range of strain at the higher concentrations of Si, Al and Y. The dynamic strain aging occurs because interstitial impurities such as oxygen and carbon adhere to dislocations [13]. Total elongation tends to decrease because of increase of work hardening [13]. In the case of a high concentration of additional elements, the temperature range of dynamic strain aging is shifted toward higher temperatures because diffusion of interstitial impurities in the materials is restrained by interaction with substitutional elements. In addition, the upper temperature limit for dynamic strain aging was observed in this experiment to decrease with decreasing concentration of interstitial impurities. Consequently, it is considered that dynamic strain aging is suppressed and the elongation is larger at 923 K as the concentration of the additional elements is increased. At 1123 K, a considerable influence of the concentration of the additional elements is observed in the curves. The deformation mode seems changed into a high temperature type as the concentration of the additional elements is higher. Therefore, the ultimate tensile strength decreases and the total elongation increases at this temperature with increasing Si, Al and Y.

The dependence of yield stress on the concentration of additional elements at 300, 923 and 1123 K is shown in Fig. 2. While the concentration of the additional



Fig. 1. Typical stress–strain curves of V–4Ti–4Cr type alloys tested at 300, 923 and 1123 K.

elements has no serious effect on yield stress, a slight increase in yield strength with increasing concentration of additional elements is observed. Although the Cr



Fig. 2. Dependence of yield stress on the concentration of Si, Al and Y tested at 300, 923 and 1123 K.

concentration of the two V–5Ti–5Cr type alloys, i.e. (g) and (h), differ by about 0.9%, the measured yield stress is not different. Therefore, it is considered that Ti mainly contributes to the difference in yield stress from these alloys.

The dependence of total elongation on the concentration of additional elements at 300, 923 and 1123 K is shown in Fig. 3. At 300 K, the total elongation ranges from 25% to 30%, with no significant difference between the alloys. At 923 K, a decrease in total elongation is observed when the total concentration of Si + Al + Y is below 0.6%. For alloys with less than 0.6% of Si + Al + Y concentration, the total elongation is about 20–25% at 923 K. At 1123 K, the total elongation increases with increasing concentration of additional elements for V–4Ti–4Cr type alloys. The total elongation of the V–4Ti–4Cr–0.5Si–0.5Al–0.5Y alloy (a) is more than 60%.

Fig. 4 shows the dependence of yield stress, ultimate tensile strength and total elongation on test temperature for the V-4Ti-4Cr type alloys. The yield stress decreases monotonously with increasing test temperature. The ultimate tensile strength is about 400-450 MPa for all alloys at temperatures up to 923 K. However, it decreases slightly for the alloys with less than 0.3% Si at 723 K. At 1123 K, it decreases evidently for all alloys and the degree of decrease is larger as the concentration of additional elements is higher. Total elongation is almost the same for the various alloys at temperatures up to 723 K. Some differences appear gradually from 923 K and total elongation is larger as the concentration of additional elements is higher. At 1123 K, the difference in the concentration of additional elements is observed conspicuously. Smith et al. reported results of tensile tests with V-4Ti-4Cr and V-5Ti-5Cr alloys at temperatures from 296 to 923 K. In that report, total elongation had the same tendency as the results in this paper while yield stress was not significantly different for V-4Ti-4Cr and V-5Ti-5Cr alloys [14].



Fig. 3. Dependence of total elongation on the concentration of Si, Al and Y tested at 300, 923 and 1123 K.



Fig. 4. Dependence of tensile properties on test temperatures for V-4Ti-4Cr type alloys. The symbols are shifted sideways in the figure for clarity; tensile tests of all alloys were performed actually at 300, 723, 923 and 1123 K.

# 3.2. Fractography

Fig. 5 shows the fracture surfaces of V-4Ti-4Cr (f) and V-4Ti-4Cr-0.5Si-0.5Al-0.5Y (a) alloys tested at 300, 723, 923 and 1123 K, respectively. Ductile dimple fracture mode is observed for both alloys at all test

temperatures. The dimple size, however, is different slightly and is inclined to be larger as the concentration of additional elements is higher.

# 3.3. Effects of Si, Al and Y on tensile properties of V-4Ti-4Cr type alloys

Effects of small amounts of Si, Al and Y addition on tensile properties of the V-4Ti-4Cr type alloys are summarized here. Tensile strengths of the series of alloys show slight dependence on the concentration of the additional elements. Yield stress and ultimate tensile strength tend to increase with the concentration of the additional elements except when testing at 1123 K. Ultimate tensile strength becomes lower at 1123 K as the concentration of additional elements is higher, since the deformation mode shifts into a high temperature type. Total elongation increases with increasing concentration of Si, Al and Y at temperatures of 923 and 1123 K. Fracture surfaces are ductile for all alloys and at all test temperatures. Therefore, from the viewpoint of tensile properties at high temperature, it is considered that appropriate concentration of Si, Al and Y in V-4Ti-4Cr alloy should be 0.5 wt%, respectively, because of its high temperature strength and ductility.

### 3.4. Influence of helium implantation

The results of tensile tests of selected specimens after helium implantation are shown in Fig. 6. Total elongation of the specimens tested after helium implantation decreased compared to specimens without implantation at all of the testing temperatures. Although total elongation of the specimens without implantation increases significantly at higher temperature, the total elongation of the helium implanted specimens tends to decrease at



Fig. 5. SEMs of fracture surfaces for V-4Ti-4Cr and V-4Ti-4Cr-0.5Si-0.5Al-0.5Y alloys tested at 300, 723, 923 and 1123 K.



Fig. 6. Dependence of total elongation on test temperatures for helium implanted and unimplanted alloys. Helium was implanted about 50 appm with 0.02 dpa.

temperatures 923 and 1123 K. TEM observation of the helium implanted specimens indicated that some bubbles were formed both in the grain boundary and in the matrix of V-4Ti-4Cr-0.5Si-0.5Al-0.5Y (a) alloy implanted helium followed by annealing at 1123 K for 3.6 ks in vacuum. The bubbles were observed also in V-5Ti-5Cr-1Si-1Al-1Y (h) alloy [10]. Intergranular fracture mode was observed in a small fraction of the fracture surface and the sides of deformed specimen. Therefore, it is believed that the high-temperature helium embrittlement occurs at 1123 K. Generally, total elongation of V-4Ti-4Cr (f) alloy is large. However, total elongation of the alloy with the smaller concentration of additional elements is not always large. V-4Ti-4Cr-0.5Si-0.5Al-0.5Y (a) alloy has a larger total elongation than V-4Ti-4Cr-0.1Si-0.1Al-0.1Y (c) alloy. There is no significant difference between the total elongations of the V-4Ti-4Cr (f) and V-4Ti-4Cr-0.5Si-0.5Al-0.5Y (a) alloys. For yield stress of the He-implanted specimens, while the dependence on the concentration of additional elements was small at more than 923 K, the increase in the V-4Ti-4Cr-0.5Si-0.5Al-0.5Y (a) alloy was about 100 MPa larger than other alloys at 300 K.

# 4. Summary

(1) From the results of tensile tests at a temperature ranging from ambient temperature to 1123 K, significant differences in yield stress and ultimate tensile stress are not observed for concentrations of additional elements Si, Al and Y ranging from 0.1% to 0.5%. The total elongation increases with increasing concentration of additional elements for tests at 923 and 1123 K. Therefore, from the viewpoint of tensile properties at high temperature, it is suggested that relatively high concentration of Si, Al and Y about 0.5% gives the best behavior among the alloys examined in this study.

(2) High-temperature helium embrittlement was observed in the specimen tested at 1123 K after helium implantation to about 50 appm with 0.02 dpa. Of all alloys in this study, V-4Ti-4Cr-0.1Si-0.1Al-0.1Y alloy has the smallest total elongation at all the testing temperatures and V-4Ti-4Cr-0.5Si-0.5Al-0.5Y alloy has the largest yield stress at 300 K. Therefore, considering both unimplanted high temperature tensile data and He-implanted tensile data, it is considered that the appropriate concentration of Si, Al and Y is from 0.1 to 0.5 wt%, respectively.

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#### References

- H. Matsui, K. Fukumoto, D.L. Smith, H.M. Chung, W. van Witzenburg, S.N. Votinov, J. Nucl. Mater. 233–237 (1996) 92.
- [2] H.M. Chung, B.A. Loomis, D.L. Smith, J. Nucl. Mater. 212–215 (1994) 804.
- [3] B.A. Loomis, H.M. Chung, L.J. Nowicki, D.L. Smith, J. Nucl. Mater. 212–215 (1994) 799.
- [4] B.A. Loomis, O.N. Carlson, in: E. Claud (Ed.), Reactive Metals, AIME, 1958, p. 227.
- [5] M. Satou, K. Abe, H. Kayano, J. Nucl. Mater. 179–181 (1991) 757.
- [6] M. Satou, K. Abe, H. Matsui, J. Nucl. Mater. 191–194 (1992) 938.
- [7] M. Satou, K. Abe, H. Kayano, H. Takahashi, J. Nucl. Mater. 191–194 (1992) 956.
- [8] M. Satou, K. Abe, H. Kayano, J. Nucl. Mater. 212–215 (1994) 794.
- [9] M. Satou, K. Abe, H. Kayano, J. Nucl. Mater. 233–237 (1996) 426.
- [10] M. Satou, H. Koide, A. Hasegawa, K. Abe, H. Kayano, H. Matsui, J. Nucl. Mater. 233–237 (1996) 447.
- [11] M. Satou, H. Koide, A. Hasegawa, K. Abe, Sci. Rep. RITU A 45 (1997) 157.
- [12] T. Masuyama, M. Morimoto, O. Konuma, K. Abe, Proceedings of the International Conference on Evolution in Beam Applications, Takasaki, Japan, 1991, p. 729.
- [13] H. Yokoyama, K. Toma, K. Abe, S. Morozumi, Philos. Mag. 23 (1971) 1387.
- [14] D.L. Smith, H.M. Chung, B.A. Loomis, H.C. Tsai, J. Nucl. Mater. 233–237 (1996) 356.