MODERATION OF NEGATIVE OXYGEN EFFECTS BY SMALL YTTRIUM ADDITION TO LOW ACTIVATION VANADIUM ALLOYS

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In order to improve irradiation embrittlement of vanadium alloys for fusion reactors, yttrium (Y) has been added reducing the interstitial oxygen impurity. However Y addition can also degrade high-temperature strength, because Y could scavenge oxygen in solid solution, which is a strong hardening agent in vanadium alloys. In this study, the effect of Y addition and oxygen level on the mechanical properties was investigated from the view points of both the high-temperature strength and low temperature ductility. Y addition was suggested to moderate the hardening and embrittlement induced by oxygen impurity sustaining the high-temperature strength within an acceptable level.

I. INTRODUCTION

V-4Cr-4Ti alloy is considered as an attractive structural material for the first-wall and blanket components of fusion reactor systems due to their low neutron-induced activation, favorable mechanical properties at high temperatures and good compatibility with lithium coolant.[1] Low-temperature irradiation embrittlement is one of the most important issues determining operation temperature limit. The low-temperature embrittlement is known to be enhanced by irradiation defects decorated by solute oxygen impurities.

It has been reported that small addition of yttrium (Y) was effective to reduce oxygen impurity and improve ductility after neutron irradiation without severe degradation of low-temperature impact properties.[2-5] Y addition, on the other hand, can also degrade high-temperature strength, because Y could scavenge solute oxygen, which is a strong hardening agent in vanadium alloys, by a formation of Y_2O_3 (Ref. 2). The effect of Y addition must be evaluated from the view points of both the high-temperature strength and the low temperature ductility.

The purpose of the present study is to investigate the effects of Y addition and impurity O level on the mechanical properties of V-4Cr-4Ti alloy at both low and high temperature.

II. EXPERIMENTAL PROCEDURES

Previous studies with 2 kg scale melting showed that the oxygen concentration was decreased with increasing Y concentration, while workability and impact properties were degraded. The Y addition of 0.15 wt% was selected for larger scale melting from a trade-off between oxygen level and impact properties.[4] V-4Cr-4Ti alloy and V-4Cr-4Ti-0.15Y alloy with different oxygen concentration were used in this study. Results of chemical analysis is shown in Table I. V-4Cr-4Ti-0.019O is the reference V-4Cr-4Ti alloy, NIFS-HEAT-2, which was fabricated by electron beam melting and vacuum arc re-melting process in 166 kg scale.[6, 7] Other alloys were fabricated by a levitation melting process in 15 kg scale.[2] The sheet specimens for Vickers hardness tests with 1 mm in thickness were annealed at 500~1000 ^{o}C for 3.6 ks after cold rolling.

TABLE I.
Chemical composition of the alloys (wt%).

Code	Cr	Ti	Y	C	N	O
V-4Cr-4Ti-0.019O	4.11	4.15	<0.002	0.025	0.009	0.019
V-4Cr-4Ti-0.051O	4.40	4.51	<0.002	0.014	0.015	0.051
V-4Cr-4Ti-0.15Y-0.011O	4.51	4.59	0.09	0.011	0.013	0.011
V-4Cr-4Ti-0.15Y-0.27O	3.87	3.99	0.06	0.010	0.018	0.27

The hardness was measured with a load of 500 gf for 30 s. Miniature tensile specimens with a gauge size of 1.2 (width) x 5 (length) x 0.25 mm (thickness), called SSJ type, were used for tensile tests.[8] Miniaturized Charpy specimens were prepared with a dimension of 1.5 mm (thickness) x 1.5 mm (width) x 20 mm (length), a notch angle of 30^{o}, and a notch depth of 0.3 mm, so that ligament size was 1.2 mm. The tensile specimens and the Charpy specimens were annealed at 1000 ^{o}C for 7.2 ks for the V-4Cr-4Ti type alloys, and at 950 ^{o}C for 3.6 ks for the V-4Cr-4Ti-0.15Y type alloys. Tensile tests were carried out at temperatures ranging from room temperature to 800 ^{o}C in a vacuum better than 1 x 10^{-4} Pa. The initial strain rate was 6.67 x 10^{-4} s^{-1}. Charpy impact tests were conducted at the temperature from liquid nitrogen temperature to room temperature by using an instrumented Charpy impact test machine in a hot cell at the Oarai Branch, Institute for Materials Research, Tohoku University. The crosshead speed of Charpy impact tests were 5 m/s.

III. RESULTS

Figure 1 shows hardness recovery as a function of the final annealing temperature after cold rolling. In both cases with and without Y, hardness for alloys with higher oxygen levels was higher than that with lower oxygen levels throughout the annealing temperature from 500 to 1100 ^{o}C. Above 900 ^{o}C hardness for V-4Cr-4Ti-0.15Y-0.27O was smaller than that for V-4Cr-4Ti-0.051O. Figure 2 plots data for the annealing temperature of 1000 ^{o}C in Fig. 1 as a function of oxygen concentration. Vickers hardness increased with increasing oxygen concentration. The slope for the trend line for V-4Cr-4Ti alloy and V-4Cr-4Ti-0.15Y alloy was 0.070 Hv/wppm O and 0.006 Hv/wppm O, respectively. The figure suggests that the hardening by oxygen is suppressed by Y addition.

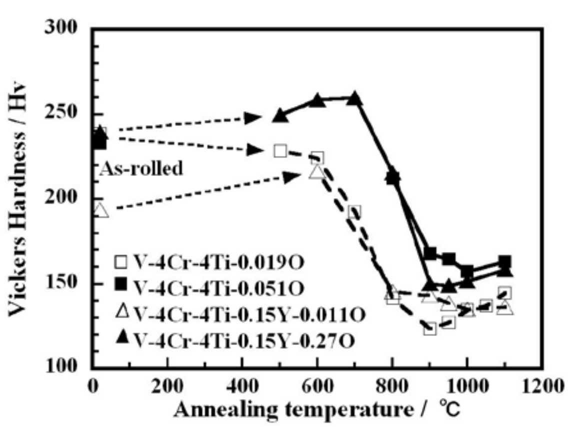

Fig. 1 Recovery of hardness as a function of final annealing (500-1100 ^{o}C) after cold rolling.

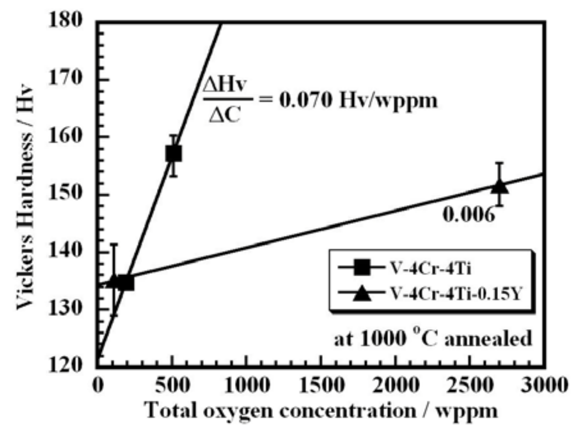

Fig. 2. Dependence of Vickers hardness on oxygen concentration. Final annealing condition was 1000 ^{o}C for 3.6 ks. $\Delta Hv/\Delta C$ means the hardening coefficient per unit oxygen concentration.

The main features of the tensile curves for V-4Cr-4Ti-0.15Y-0.27O are illustrated in Fig. 3, which contains data from room temperature to 800 ^{o}C. At the temperatures between 300 and 600 ^{o}C, serrations were observed whose amplitude and strain range of appearance varied with temperatures. Figures 4 and 5 show tensile curves for the four alloys at 600 and 700 ^{o}C, respectively. Serrations for V-4Cr-4Ti-0.15Y alloys disappeared at 700 ^{o}C as shown in Fig. 5. No serration was observed above 750 ^{o}C for all the alloys.

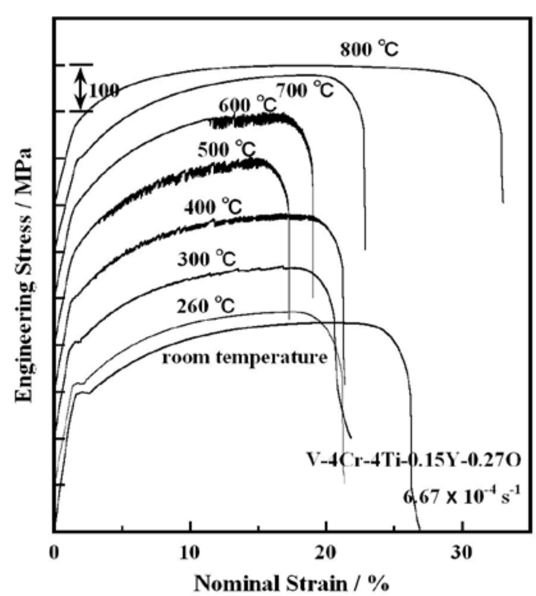

Fig. 3. Tensile curves for V-4Cr-4Ti-0.15Y-0.27O.

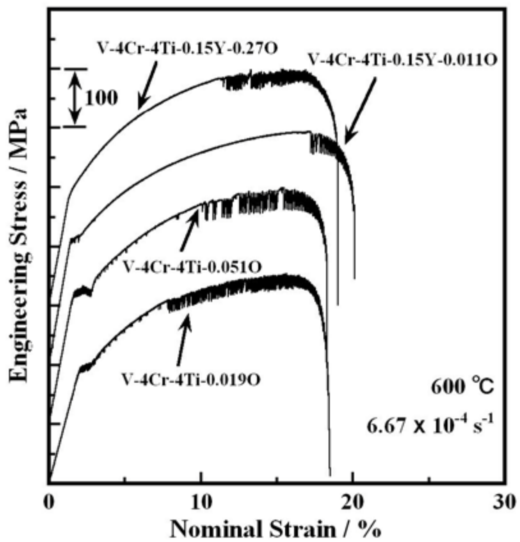

Fig. 4. Tensile curves for the alloys at 600 °C.

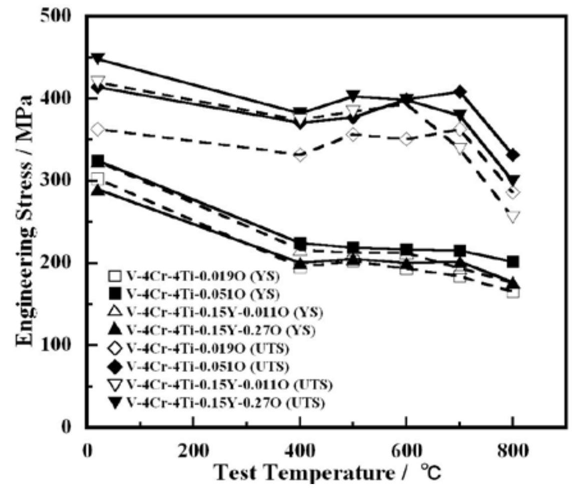

Fig. 6. Dependence of tensile strength on test temperatures.

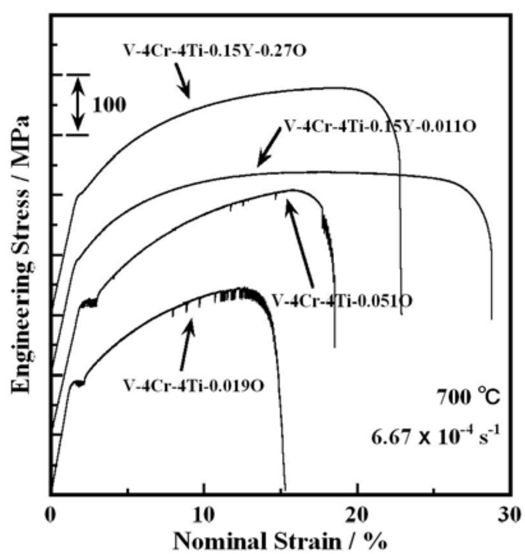

Fig. 5. Tensile curves for the alloys at 700 °C.

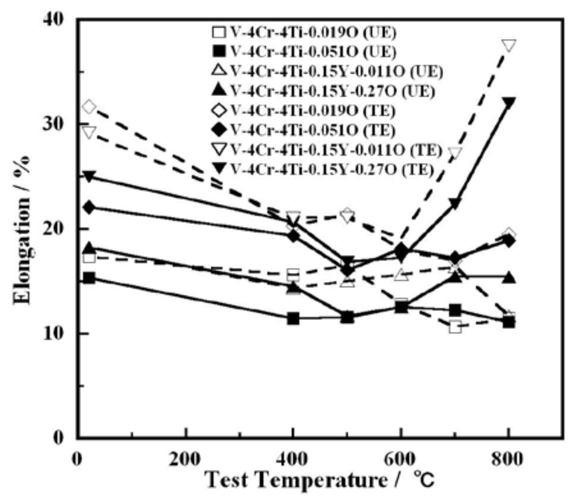

Fig. 7. Dependence of elongation on test temperatures.

Figures 6 and 7 show the dependence of tensile strength and elongation on test temperature, respectively. Between 600 to 700 °C, ultimate tensile strength (UTS) for V-4Cr-4Ti alloys increased, while that for V-4Cr-4Ti-0.15Y alloys decreased. UTS at 700 °C was decreased with Y addition. The reduction of yield stress (YS), however, was smaller than that of UTS. Total elongation (TE) increased above 700 °C for V-4Cr-4Ti-0.15Y alloys. Decrease in strength and increase in elongation at 700 °C for V-4Cr-4Ti-0.15Y alloys are likely to correspond to the loss of serration, as shown in Figs. 4 and 5.

Figure 8 shows results of Charpy impact tests. The absorbed energy for V-4Cr-4Ti-0.15Y-0.27O alloy above -100 °C was comparable to that of V-4Cr-4Ti-0.019O alloy, and was evaluated to be 0.4 J mm^{-3} as its upper shelf energy (USE). Therefore ductile-brittle transition temperature, DBTT, for V-4Cr-4Ti-0.15Y-0.27O was estimated to be around -196 °C. This is sufficiently low as a structural material. Therefore, the increase in oxygen level to 0.27 wt% did not degrade impact properties significantly for the alloy with Y addition.

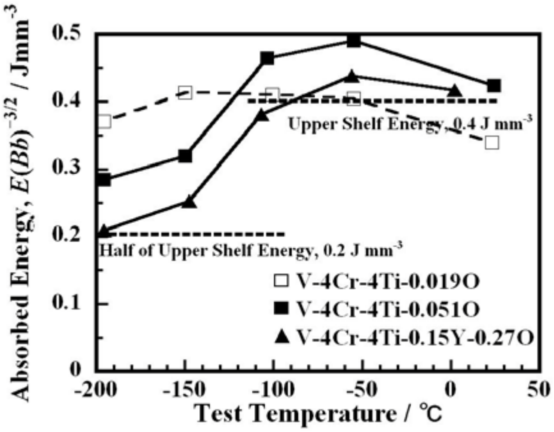

Fig. 8. Absorbed energy in Charpy impact test for the alloys. Absorbed energy is normalized with specimen width, B = 1.5 mm, and ligament size, b = 1.2 mm.

IV. DISCUSSION

Figure 2 shows that the increment in hardness by the increase in oxygen level was suppressed by Y addition. In the previous study, precipitations of Y_2O_3 were observed in the Y-added alloy.[2] Oxygen concentration in solution is considered to be reduced by the formation of Y_2O_3. Thus Y addition can moderate solid-solution hardening by oxygen.

Serration behavior varied with the temperature as shown in Fig. 3. The serration occurs when the impurity in solution interacts with the moving dislocations during the deformation. The temperature where serrations disappeared for V-4Cr-4Ti-0.019O (750-800 ^{o}C) was consistent with that of Ti-CON formation.[9] Ti-CON precipitation might decrease impurity concentration in solution and eliminate serrations. On the other hand, serrations for V-4Cr-4Ti-0.15Y-0.27O disappeared at 700 ^{o}C. The growth of Y_2O_3 precipitates could occur at lower temperature than that of Ti-CON formation, due to stronger affinity between Y and oxygen.[10] Therefore Y suppresses serrations at 700 ^{o}C and degrades high-temperature strengths.

Thermal and structural analyses on Li cooling channels in fusion blanket have given the maximum equivalent stress as 56 and 35 MPa for a stainless steel and a vanadium alloy, respectively.[11, 12] According to these examples, the design stress required for vanadium alloys will be several 10 to around 100 MPa. The YS and UTS at 700 ^{o}C for V-4Cr-4Ti-0.15Y-0.011O alloy were 194 MPa and 339 MPa, respectively. The YS (UTS) is 5.5 times (9.7 times) larger than the equivalent stress for the vanadium alloy (35 MPa), and 1.9 times (3.4 times) larger than the expected design stress (100 MPa). Though the design criteria and safety factor to the design stress have not been determined for fusion structural materials, the above margins are considered to be still enough as the safety factor to blanket design. Especially for V-4Cr-4Ti-0.15Y-0.011O alloy, the reduction in UTS by Y addition was 25 MPa, however, is thought to be acceptable for the application to structural materials.

V. SUMMARY

The effect of Y addition on mechanical properties has been clarified from the view points of both the high temperature strength and the low temperature ductility.

Y addition suppressed the hardening by oxygen. With Y addition, oxygen increase to 0.27 wt% did not degrade impact properties. Thus, Y addition moderated the hardening and embrittlement induced by oxygen impurity to a high level of 0.27 wt%.

Y addition can suppress serrations in the tensile tests at 700 ^{o}C. Y is considered to reduce solute oxygen by the formation of Y_2O_3 and to suppress serrations as a result. The reduction in high-temperature strengths corresponds to the suppression of serrations. With Y addition, the UTS was reduced and the elongation was improved above 700 ^{o}C. The reduction in UTS by Y addition, however, was as small as 25 MPa at 700 ^{o}C, which is thought to be acceptable for the application to structural materials.

ACKNOWLEDGMENTS

The author is grateful to Mr. M. Yamazaki in the Oarai branch, Institute for Materials Research, Tohoku University, for supports in the impact tests, and to a financial support of Research Foundation for the Electrotechnology of Chubu.

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