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Fracture properties of high-purity V–4Cr–4Ti alloy (NIFS-HEAT-2) at room temperature

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Abstract

A high-purity vanadium alloy, V–4Cr–4Ti, NIFS-HEAT-2, was produced using electron beam melting and the vacuum arc re-melting process. The impurity was reduced to be as small as possible and a 26 mm thick plate was fabricated successfully. The normal size specimens were machined out of the plate to investigate the mechanical properties. Tensile tests and fracture toughness tests were conducted in air and in vacuum (about 10^{-4} Pa) environment at room temperature. The alloy showed high ductility and clear dimple fracture occurred in both environments. A ductile crack growth was not observed during the fracture toughness tests. The crack resistance curve went up straightly, and it was almost the same as the blunting line of which slope was $2 \times \sigma_{flow}$. The blunting continued over several hundreds of J value in MJ/m^2. The material is very tough and the valid J is not obtained at room temperature using a 12 mm thick compact tension specimen.

1. Introduction

Vanadium alloy is one of the candidates of the structural material for in-vessel use in a fusion device, and expected having lower decay period after irradiation. Some studies on the alloy have been carried out [1–5] and useful knowledge has been obtained. At National Institute for Fusion Science (NIFS), an attempt to produce the vanadium alloy in engineering scale has been performed under the collaboration among Japanese Universities [6]. Two 80 kg ingots of the alloy were produced and 26 mm thick plates were fabricated to certify the productivity of engineering size plates. Metallurgical studies have been reported in Ref. [7].

As the vanadium alloy is supposed to support in-vessel equipments, such as the first wall and divertor structures, in a plasma vacuum vessel, the productivity of the plate must be known and the mechanical properties of an engineering size plate must be clarified and piled up as a data base. Therefore, an attempt to fabricate the thick plate was performed, and the tensile and the fracture toughness tests were conducted using the standard size specimens in air and in vacuum at room temperature. This report presents the test results, and the mechanical aspect of the alloy is discussed through the tests in comparison with Heat 832665 and 832864 V–4Cr–4Ti alloy [2,3].

2. Test material and experimental procedure

The material tested in this study is a vanadium alloy, which has been newly developed in NIFS. It contains the following chemical compositions in wt%: 4.07Cr, 3.88Ti, 0.0052H, 0.0050C, 0.0089N, 0.0178O. The alloy was fabricated by electron beam melting and a vacuum arc re-melting process. The ingot was canned with 304 stainless steel by electron beam welding. The canned ingot was hot-isostatically pressed with a pressure of 103 MPa at 1463 K for 4 h, then hot-forged at 1173–1473 K. The forged ingot was 76% cold-rolled into a 26 mm thick plate after removing the can. A niobium box containing the alloy plate was wrapped with a zirconium foil and annealed in a vacuum of 6–12 × 10^{-4} Pa at 1273 K for

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2 h. The microstructure is shown in Fig. 1. The average grain size is 30–40 μm. The oxygen content is almost half, comparing to Heat 832665 and 832864 produced by US Department of Energy and General Atomics, respectively [3].

The tensile test specimen had a 30 mm long parallel part with a 6.25 mm diameter and M12 threads on both ends. The total length was 75 mm. The shoulder radius on the end of the parallel part was 10 mm. The specimen was machined out at 1/4 position of the plate thickness and kept in a conventional vacuum container. Before the test, the diameter and marker distance of about 27 mm were measured to evaluate reduction in area (RA) and elongation (El). Two extensometers, of which sensitivity was about 5000×10^{-6} strain/mm and the gage length was 6 mm, were attached to the parallel part of the specimen symmetrically to measure Young's modulus and yield stress, canceling a bending component. A stroke rate of 0.75 mm/min was adopted and tests were done in air and in vacuum of about 1×10^{-4} Pa at room temperature. Outputs of the load, stroke and extensometers were stored in a computer at the rate of 10 samples per second.

The fracture toughness test was carried out using a 12 mm thick compact tension (CT) specimen as shown in Fig. 2. The specimen was taken out at 1/4 position of the plate by electro-discharge machining. The initial notch was machined to make the crack propagate in the transverse direction to the rolling direction (L–T orientation). Since the pre-crack was induced in vacuum and sequentially the fracture toughness test was performed to avoid the effect of air on the toughness in vacuum, no side groove was machined. Before setting up the specimen, the surface was polished by emery paper up to #1500 without water. Then markers were scratched to measure the crack length.

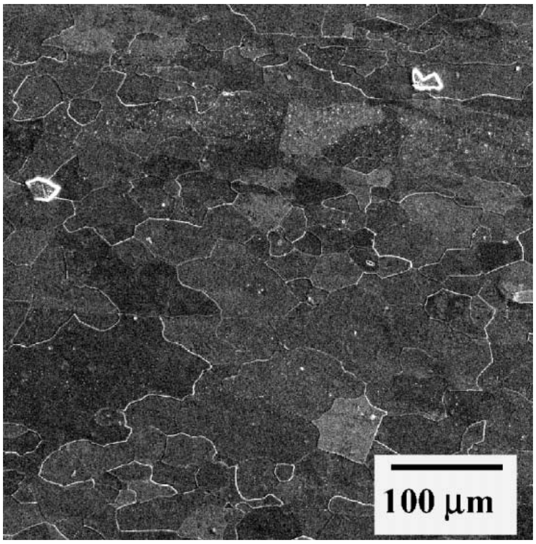

Fig. 1. Microstructure on rolling-thickness plane.

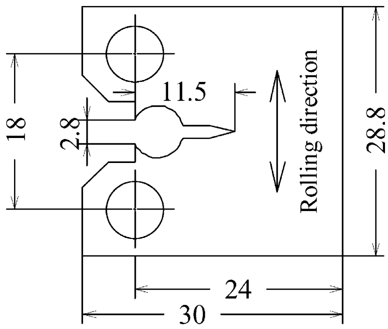

Fig. 2. CT specimen.

The specimen was set up in a vacuum chamber equipped with a servo-controlled fatigue testing machine of which capacity was 98 kN. The fatigue pre-crack was induced in air for the test in air and followed by the fracture toughness test in air. During the fatigue process, the compliance (ratio of mouth crack opening displacement (COD) to load) was monitored to evaluate the crack extension. The crack length on the polished surface was measured in parallel using the scratched markers. After the crack extension, the pre-crack length was determined carefully, and the fracture toughness test was performed by a single specimen method [8]. The stroke rate of 0.75 mm/min was adopted and the load and mouth COD were recorded at the rate of 10 samples per second. After the fracture toughness test, the specimen was fatigued again to make the crack front shape clear, for the obvious stable crack extension was not observed on the change in compliance during the fracture toughness test. The test procedure in vacuum was almost the same as the test in air, but with the only difference on the determination of the pre-crack length. Since the introduction of the pre-crack and the fracture toughness test were performed sequentially in vacuum, the pre-crack length was measured in air after the fracture toughness test. And the additional fatigue crack was induced in air after the fracture toughness test in vacuum. The vacuum was kept about $0.5–1.0 \times 10^{-4}$ Pa during the test.

3. Test results and discussion

3.1. Tensile test results

An example of the stress–strain curve measured by extensometers is shown in Fig. 3. Young's modulus was determined clearly and the material showed rather high elastic limit and lower strain-hardening near yield stress.

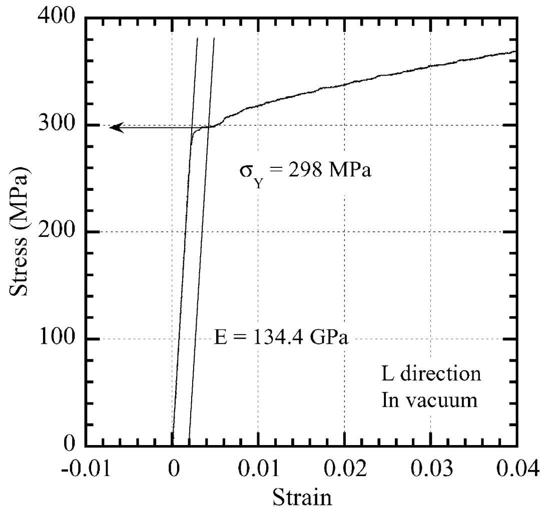

Fig. 3. Stress–strain curve of V4 specimen.

It means that the alloy behaved like a perfect elasto-plastic body. The tensile test results are summarized in Table 1. There is no remarkable difference between those in air and in vacuum. Roughly saying, the alloy has Young's modulus of 130 GPa, the yield stress of 300 MPa and the ultimate tensile strength of 400 MPa. In addition, the alloy is very ductile showing over 80% RA and 30% El. Young's modulus of the alloy is the same as that of pure vanadium and little difference is caused by alloying. Fig. 4 shows the fracture surface of V4 sample. It is clear that the necking proceeded well and dimple fracture occurred.

The tensile tests with miniature specimens of NIFS-HEAT2 are not performed. Therefore, the effect of the specimen configuration on the tensile properties is not clear and will be discussed when the irradiation tests with miniature specimens are finished. Bray et al. reported the tensile data of Heat 832864 (0.0370O, 0.0120N, 0.0030C, 0.0270Si in wt%) with a specimen of $1.52(w) \times 0.76(t) \times 7.6(l)$ mm^3 [3]. The yield stress, the ultimate tensile strength and the total El at room temperature are 315, 410 MPa and 28.5%, respectively, and the present results show the lower yield stress and the lower tensile stress and the higher El. It seems that the higher impurity content will reduce the El and small specimen configuration will increase the yield stress and the ultimate tensile strength. To confirm this consideration, further investigations are needed.

Table 1
Summary of tensile test results

Specimen #	E (GPa)	σ_Y (MPa)	σ_u (MPa)	El (%)	RA (%)	Direction	Environment
V1	–	–	–	29.7	81.9	T	Air
V2	130.1	304	417	31.2	83.1	T	Vacuum
V3	129.0	289	401	34.0	87.7	L	Air
V4	134.4	298	411	35.3	86.8	L	Vacuum

L – rolling direction; T – transverse direction.

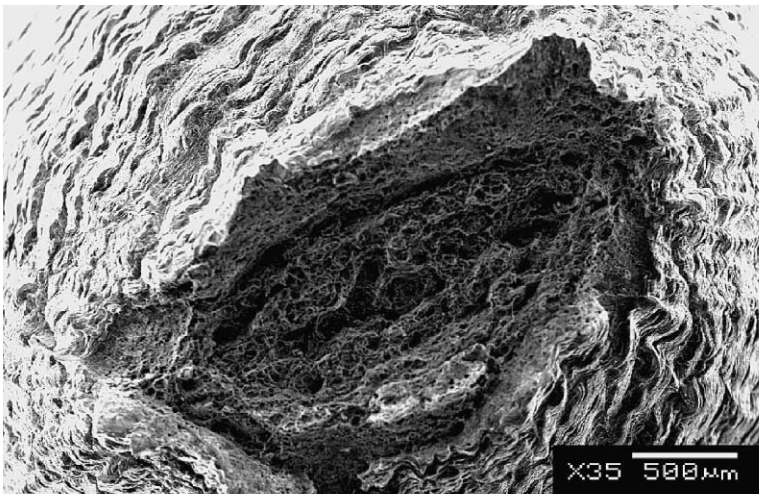

Fig. 4. Fracture surface of V4 specimen.

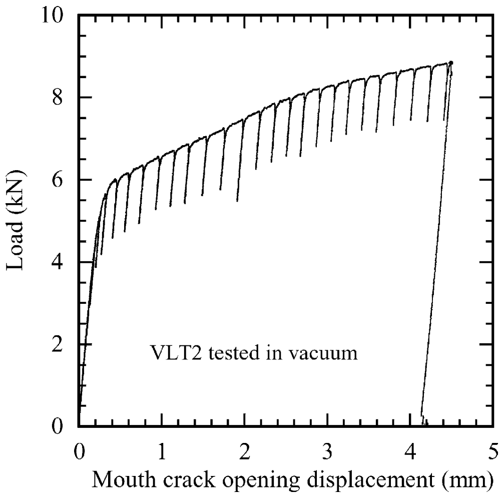

Fig. 5. Load–mouth COD curve of VLT2 specimen.

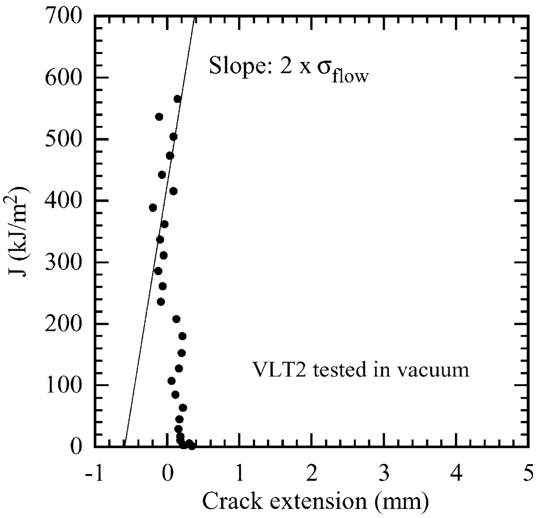

Fig. 6. *J–R* curve of VLT2 specimen.

3.2. Fracture toughness test results

The load–mouth COD curve and *J–R* curve of VLT2 specimen are shown in Figs. 5 and 6. Since VTL1 specimen tested in air showed the same tendency as VTL2 specimen, only the results of VLT2 are presented here. The mouth COD became large and strain-hardening occurred. When the mouth COD reached at about 4.4 mm which is a capacity of the clip-on gage used in the test, the output became unstable and the test was stopped. Since the specimen deformed too much, a rotational factor was taken into account when *J*-integral was calculated [8].

The crack growth resistance curve (Fig. 6) does not show the crack extension and data are still on the crack blunting line. This means that the alloy has high toughness and the ductile crack does not initiate at the stretched zone. The final value of *J*-integral exceeds the requirements of plane strain condition described in

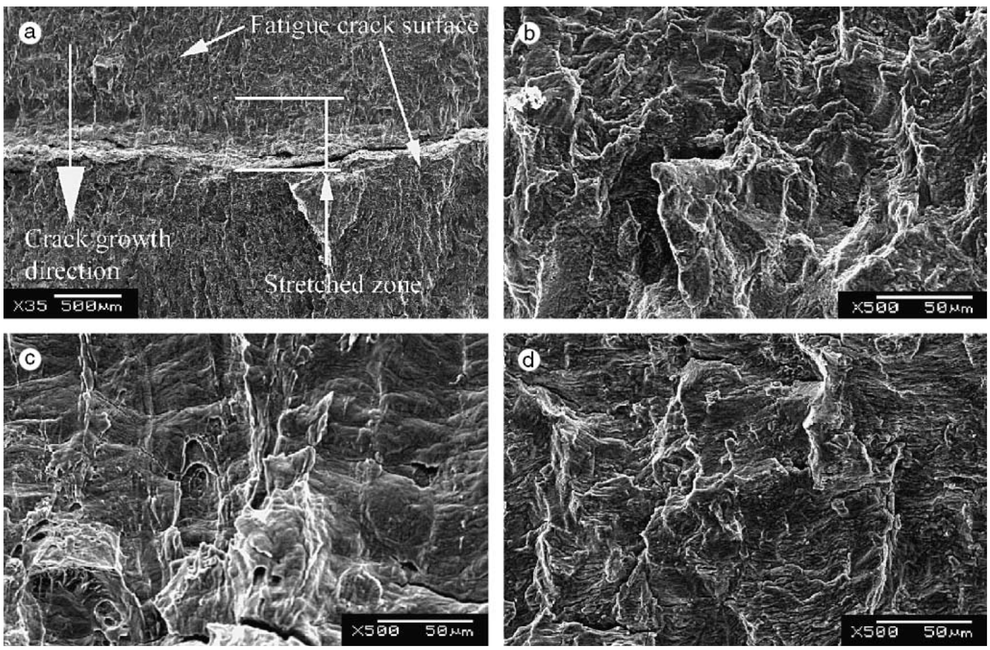

Fig. 7. (a) General view of stretch zone on VLT2, (b) near stretch zone on notch side (striations), (c) enlarged view of stretch zone and (d) upper terrace near stretch zone (striations).

ASTM standard. The same behavior was observed in Heat 832665 [2].

The fracture toughness is considered to be one of the material properties and is constant under plane strain condition. And the valid toughness, which satisfies the requirements described in ASTM standard, will be obtained with increasing the specimen thickness. However, the actual thickness of vanadium alloy plate applicable to the real structure in the fusion device is limited and it is useful to clarify the fracture behavior of the engineering thick plate for the design of the structure. From the view point of such an engineering sense, it must be noted that the 12 mm thick plate of NIFS-HEAT-2 has remarkable resistance to the ductile crack initiation under Mode I fracture mode.

The SEM observation results are shown in Fig. 7. Fig. 7(a) shows the low magnification image on the stretched zone at the center of VLT2 specimen. The notch root side surface on the stretched zone is magnified and shown in Fig. 7(b). The surface is formed with a fatigue surface. Fig. 7(c) shows the magnified image of stretched zone. The ductile surface and dimples are observed. On the top of the terrace, many striations were formed. As the additional fatigue loading was applied after the fracture toughness test, it is good evidence that no slow crack growth occurred during the toughness test. It supports the consideration on J–R curve.

Li et al. showed the brittle delaminations in planes normal to the thickness direction on the fracture surface of the CT specimen [2]. However, the both specimens tested in air and in vacuum did not show such brittle delaminations and whole surface was ductile.

4. Conclusions

The tensile and the fracture toughness tests were carried out using the 6.25 mm diameter round bars and the 12 mm thick CT specimens to characterize the high-purity V–4Cr–4Ti alloy, NIFS-HEAT-2. The main results are summarized as follows:

(1) The alloy shows good ductility both in air and in vacuum at room temperature in the tensile test.
(2) Also, stable necking progresses well and the final fracture is caused by dimple formation.
(3) The fracture toughness tests show that the alloy is very tough in air and in vacuum at room temperature.
(4) And a ductile crack does not initiate at the stretched zone both in air and in vacuum.

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