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Irradiation hardening of V–4Cr–4Ti [1]

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Abstract

In the framework of the European Long Term Fusion Technology Program, Advanced Materials Field, ECN has been working on the assessment of low temperature irradiation hardening and embrittlement of vanadium alloys, as being developed for fusion application. Tensile, miniaturized Charpy impact (KLST) and Compact Tension specimens have been irradiated in the High Flux Reactor (HFR) in Petten up to approximately 6 dpa at 600 K. Three alloys were included; V–4Cr–4Ti from the 500 kg IEA reference heat provided by Argonne National Laboratory, and minor amounts of V–3Cr–3Ti and V–6Cr–6Ti, provided by Oak Ridge National Laboratory. The paper presents the results of tensile tests after irradiation. These tensile tests show strong hardening and reduction of ductility.

1. Introduction

In the framework of the European Long Term Fusion Technology Program, ECN has been working on the assessment of low temperature irradiation hardening and embrittlement of vanadium and ferritic/martensitic alloys, as being developed for fusion application [1–4]. Vanadium alloys are considered to be an option for the structural material for future thermonuclear fusion reactors. One of the major drawbacks of vanadium alloys is, as it is for ferritic/martensitic alloys, low temperature irradiation embrittlement. Therefore, tensile, miniaturized Charpy impact (KLST) and Compact Tension specimens have been irradiated in the High Flux Reactor (HFR) in Petten up to approximately 6 dpa at 600 K. Three alloys have been investigated, mainly V–4Cr–4Ti and minor amounts of V–3Cr–3Ti and V–6Cr–6Ti. Post-irradiation mechanical testing was to be completed in 1998. The results of the completed tensile tests are reported in this paper.

2. Experimental procedure

2.1. Materials and specimens

Three vanadium alloys have been investigated. Two of them, V–3Cr–3Ti and V–6Cr–6Ti, were small experimental laboratory heats. The third was the TWCA produced IEA fusion reference alloy V–4Cr–4Ti, heat 832665 [5]. Two material conditions of the latter have been used in this work: TWCA rolled product-form with final heat treatment of 2 h at 1000°C, and as-received rolled product-form with additional recommended heat treatment of 1 h at 1000°C (encapsulated in quartz capsules filled with pure argon after high vacuum evacuation). The laboratory heats were both received in the final heat treated state of 2 h at 1000°C.

The cylindrical tensile test specimens have a diameter of 4 mm and a gage length of 20 mm. The V–4Cr–4Ti specimens were manufactured in the L (rolling) orientation.

2.2. Irradiation experiment

The tensile specimens were irradiated in the mixed spectrum reactor HFR, in a capsule of the type ILAS, containing 56 tensile specimens, four neutron flux monitors and four gamma-scanwires.

The specimens were in contact with a helium/neon mixture that served as the cooling medium. The

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temperature of the capsule was controlled by adjusting the concentration of helium. The temperature was monitored by thermocouples in the drum; the corresponding temperature of the specimens was calculated by means of a finite element model. Later versions of ILAS irradiation capsules included several thermocouples inserted into specimens; the correspondence of the FEM model calculations with the measurements was found to be reasonably good.

The dose level of each specimen was calculated by inter- or extrapolation of the results of the measurements of the neutron monitor-sets and the gamma-scan wires [6]. The dose levels were found to range from 4 to 6 dpa, the variation being caused by the flux profile across the capsule. A cross-section of 1.74×10^{-25} m^{-2} was used for the calculation of the dpa rate for vanadium. The formation of helium, mostly due to the boron-content of the material, was calculated prior to the irradiation to be about 5 appm [7] (no actual helium content measurements are available yet). Thus the final He/dpa ratio of the irradiated material was approximately 1. The activation of the material was also calculated, taking into account the irradiation period and irradiation position, resulting in 2 TBq/kg after 3 months after the end of radiation. This is very low compared to many other "low activation" materials, currently under investigation. The chromium formation by the thermal neutron reaction with vanadium, which could be important because of additional embrittlement [8,9], was calculated to be 0.7% [7]. A further check on the effect of chromium formation was planned by means of a spectrally tailored irradiation experiment which started in October 1997.

2.3. Tensile tests

Tensile tests were performed in the ECN Hot Cell Laboratory. Tests were performed at temperatures from 300 K (room temperature) to 700 K. The specimens were tested in air. Heating up and stabilising of the temperature can take up to one and a half hour. Load–displacement curves were recorded by computer during the tests. These were transformed to engineering stress–strain curves to determine, for example, yield stress, ultimate tensile strength and uniform (plastic) elongation. By measuring the broken specimens after testing the fracture elongation, the reduction of area was determined. The total elongation, or fracture elongation, is measured after testing by fitting the two halves. Strain-rate was 5×10^{-4}/s, unless noted otherwise.

Several tests were analysed to examine the true stress–true strain behaviour. Up to maximum load the recorded engineering stress–strain data were used, beyond maximum load a few distinct points were obtained by measuring the reduction of area of the necked region. The true stress data were not corrected for triaxiality.

3. Results

3.1. Tensile test curves at 600 K

Two typical tensile curves for an unirradiated and an irradiated specimen tested at 600 K are presented in Fig. 1. The unirradiated specimen showed typical yield point behaviour; an upper yield point was found followed by a drop to the lower yield point, followed by a more or less constant load during yield elongation before further strain hardening occurs. The yield stress (YS) wherever used in this paper is in fact the lower yield value (plateau value). All materials showed this characteristic discontinuous yield behaviour in the unirradiated state.

The irradiated specimens showed a completely different behaviour. There was still a yield peak, but no plateau followed or strain hardening occurred, the engineering stress decreased monotonously with increasing strain until fracture. Another thing to note was the enormous amount of irradiation hardening; the yield stress increased four-fold and it coincided with the ultimate tensile strength. The resulting uniform elongation was negligible, and total elongation was reduced by a factor of three. It should be emphasised that in spite of virtually zero uniform elongation, there remains considerable ductility: 8% total elongation and 60% reduction of area (compared to respectively 25% and 90% for unirradiated specimens). Finally, it was observed that the irradiated specimens fail at a much higher engineering fracture stress than the unirradiated specimens (530 and 130 MPa respectively). The failure mode of the irradiated specimens tested at 600 K was ductile dimple fracture.

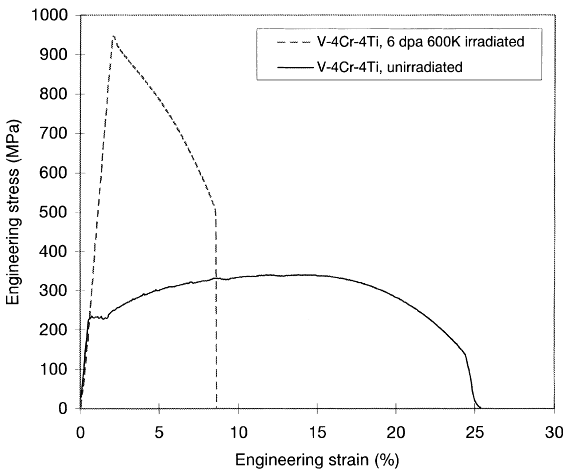

Fig. 1. Typical tensile test curves of an unirradiated and a 600 K, 6 dpa irradiated specimen. Test temperature is 600 K.

3.2. Tensile properties as function of test temperature

In Fig. 2(a) and (b) the tensile properties were presented as a function of the test temperature, for both unirradiated and irradiated material. The tensile stress decreases with increasing test temperature for the irradiated material, coming down from approximately 1100 MPa at room temperature to 950 MPa at 600 K. The irradiated and non-irradiated state have an identical temperature-trend: 150 MPa decrease over temperature range 300–600 K. The irradiated material shows no further significant additional decrease of hardening (by annealing) up to 700 K (100 K above irradiation temperature). The total elongation (TE) shows the characteristic trend for non-irradiated material: the room temperature TE was higher than at elevated temperature. On the contrary, the irradiated material shows lowest ductility at room temperature. In these graphs, the differences between the three alloys can also be observed: the V–6Cr–6Ti shows somewhat lower YS and UTS and higher TE and RA after irradiation than the other two alloys; the V–3Cr–3Ti shows lowest TE and RA. Note that only one specimen each of V–3Cr–3Ti and V–6Cr–6Ti was tested at 600 K.

In Fig. 2(a) and (b) the variation of dose level among the specimens was not taken into account; part of the observed scatter can be attributed to this, as will be shown in the next section. Another source of scatter was a small difference in tensile properties of the 1 h annealed material and the as-received material, which had been annealed for 2 h – in these plots the data points of the two were taken together. As the small variability of tensile properties in the unirradiated state hardly indicates difference in tensile properties between the two material conditions, irradiation magnifies the variability, the 2 h annealed material clearly showing the lower TE and RA.

The RA shows no temperature dependency in the non-irradiated state; after irradiation it was lowest at room temperature and higher at elevated temperature. The RA of irradiated V–4Cr–4Ti shows a maximum at approximately 600–650 K. Also, the RA was not reduced as dramatically by irradiation as the UE or TE. Even larger variability was present in the irradiated RA data, particularly at room temperature. The increase of the RA variability at room temperature was accompanied by occasional appearance of cleavage facets on the fracture surfaces.

All irradiated specimens showed indications of similar upper yield point characteristics as for the unirradiated state, except the irradiated V–6Cr–6Ti which looses its upper yield characteristic and has the lowest yield values of all irradiated materials (and highest in the unirradiated state). Thus the irradiation hardening was significantly different in V–6Cr–6Ti material. The irradiated V–3Cr–3Ti shows the lowest TE and RA.

3.3. Tensile properties as a function of dose level

There was a variation of dose level along the vertical axis of the irradiation capsules and this provided a limited dose level dependence study. The dose level dependence of YS and ductility (RA and TE) just below 6 dpa turns out to be weak but present, as can be seen in Fig. 3. (the drawn curves serve only as eye guides and do not intend to indicate the process of the intermediate build up of radiation damage). This means that the irradiation hardening and embrittlement has almost but

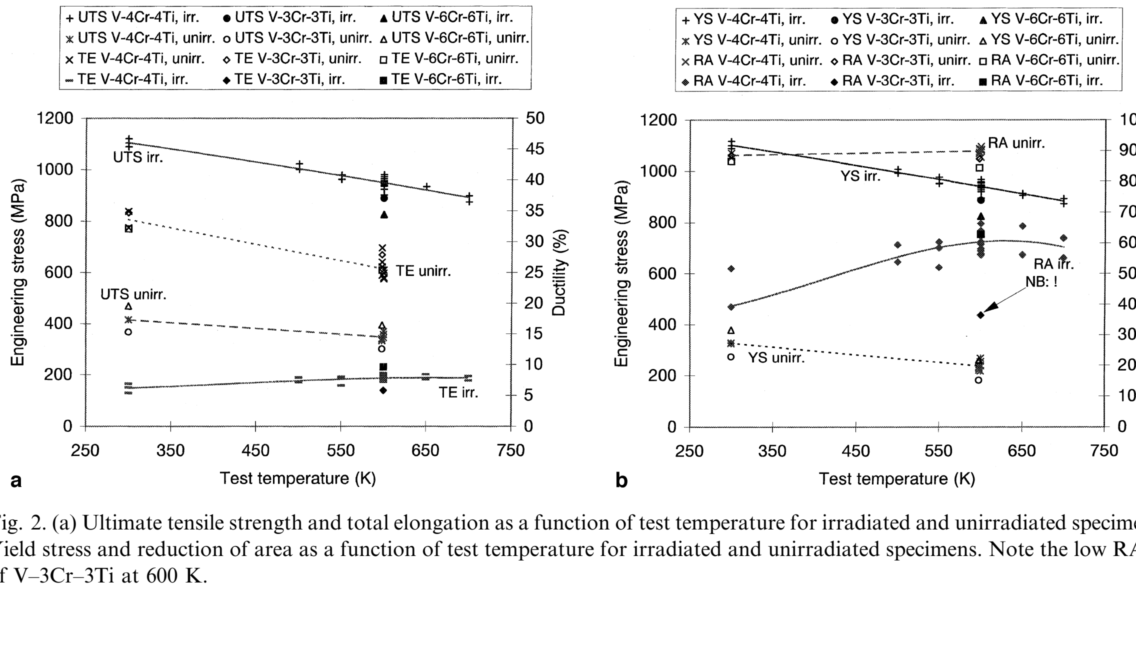

Fig. 2. (a) Ultimate tensile strength and total elongation as a function of test temperature for irradiated and unirradiated specimens. (b) Yield stress and reduction of area as a function of test temperature for irradiated and unirradiated specimens. Note the low RA value of V–3Cr–3Ti at 600 K.

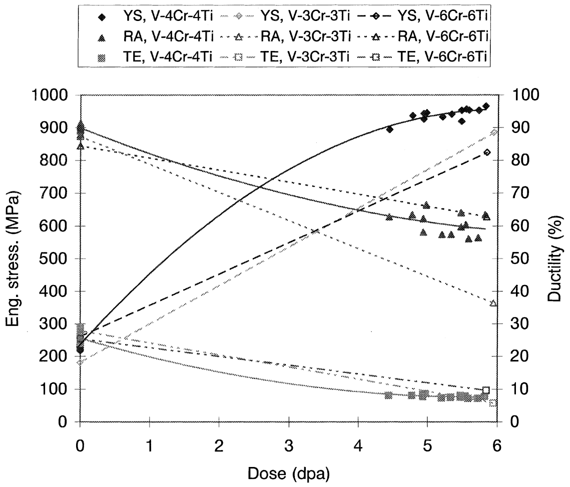

Fig. 3. Dependence of YS, RA and TE on irradiation dose for V–4Cr–4Ti. Tested and irradiated at 600 K. The drawn curves serve only as eye-guides.

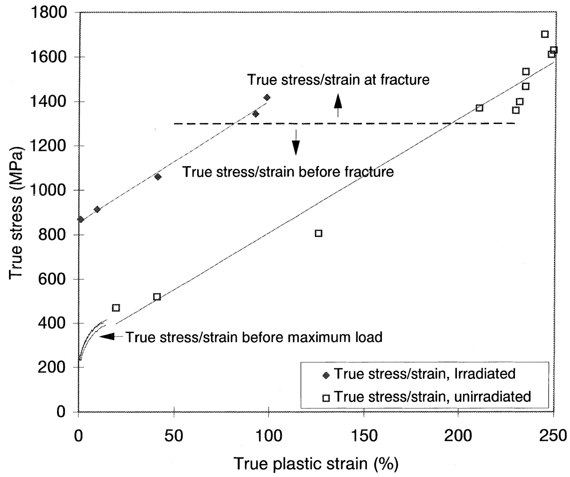

Fig. 4. True stress–strain necking behaviour of unirradiated and irradiated V–4Cr–4Ti, tested at 600 K. The stress has not been corrected for triaxiality.

not yet completely saturated and will probably continue to build up a little more with further dose increase.

3.4. True stress–strain behaviour

The irradiated material shows virtually no (engineering) strain hardening, but considerable ductility remains. The question is what happens past maximum load: is the material (partially) strain softening or is the decrease in engineering stress simply caused by necking of the specimen. To investigate this, several interrupted tensile tests were performed; i.e. the test was stopped at certain strain value, past maximum load but before fracture. Subsequently the reduction of area of the necked region was measured, and the true stress was calculated (in terms of load divided by area, no corrections for curvature/triaxialy were done). The tests were performed at 600 K, up to engineering strains of 0.6%, 1.6% and 5.0% for irradiated specimens.

The results plotted in Fig. 4 show that the true stress–strain necking behaviour for unirradiated and irradiated specimens was similar: the slopes of the linearized curves beyond maximum load were approximately parallel although the true stress values were much higher than for non-irradiated material. Necking seems to start immediately after maximum load, the observed decrease in engineering stress can be attributed to necking of the specimens.

3.5. Strain-rate effect

All tests reported above were performed at a strain-rate of 5×10^{-4}/s. A few tests were performed at lower strain-rates of 5×10^{-5}/s and 5×10^{-6}/s at a test temperature of 600 K. In general no significant effect on tensile properties was found, except for the RA of the V–3Cr–3Ti specimens. The V–3Cr–3Ti specimen tested at the conventional strain-rate 5×10^{-4}/s showed much lower RA (<40%) than the other materials, but at 5×10^{-6}/s the RA of V–3Cr–3Ti (>50%) approached the RA of the other materials (~60%). It should be noted once more that only one V–3Cr–3Ti specimen was tested at 600 K, 5×10^{-4}/s strain-rate.

3.6. Fractography

The fracture surfaces of several tensile specimens were inspected visually and by SEM. Large ellipticity of the fracture surface occurred for the unirradiated spec-

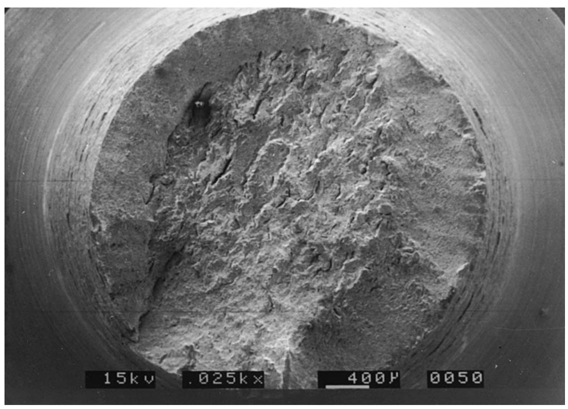

Fig. 5. SEM image of fracture surface of irradiated V–4Cr–4Ti specimen, tested at 300 K, presence of micro-sub-cracks and isolated cleavage facets.

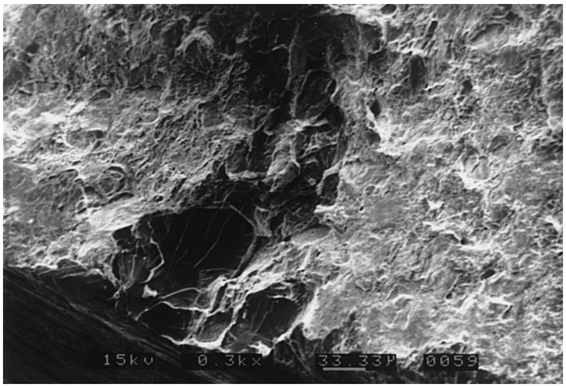

Fig. 6. SEM image of a cleavage facet of irradiated V–4Cr–4Ti specimen tested at 300 K.

imens, clearly indicating anisotropic properties. This was observed for all three alloys. This behaviour did not occur for the irradiated specimens.

The fracture surfaces of irradiated specimens, tested at 300 and 600 K showed significant differences: At 600 K a fully ductile dimple structure is observed. At 300 K the structure consists of ductile dimples and micro-sub-cracks mostly oriented parallel to each other, Fig. 5. Further, isolated cleavage facets are observed, Fig. 6.

4. Summary and conclusions

The three vanadium alloys investigated show very strong hardening after 4–6 dpa irradiation at 600 K. The hardening was lowest for V–6Cr–6Ti. The irradiation damage is close to saturation, as the dose level dependence between 4 and 6 dpa is weak.

The discontinuous yield behaviour (yield peak) of V–4Cr–4Ti and V–3Cr–3Ti was not affected by the irradiation, and additionally the temperature trend of the yield stress was not affected by irradiation; this means that short range dislocation barriers were not affected. This was not the case for V–6Cr–6Ti (does not show a yield peak after irradiation) which also shows least irradiation hardening.

The uniform deformation capacity was completely lost after irradiation, no uniform strain hardening capacity was left either. The radiation hardening was not recovered when specimens were tested at 100 K above the irradiation temperature.

The fracture mode was in general ductile, although when testing at 300 K isolated cleavage facets appear. Together with the very high yield stress at this temperature, lowest ductility and large scatter of reduction of area, this could be an indication of being near the cleavage stress.

No significant strain-rate effect was observed at 600 K, perhaps except for V–3Cr–3Ti for which the ductility is very low at the highest strain-rate.

The reduction of area was reduced by irradiation, however similar necking behaviour (in terms of true stress–strain) was shown for irradiated and non-irradiated specimens. The true stress at fracture was similar for irradiated and non-irradiated material, even though true strains differed largely.

Small differences in the ductility of V–4Cr–4Ti with different heat treatments were clearly magnified by the irradiation.

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