A MODIFIED TRITIUM TRICK TECHNIQUE FOR DOPING VANADIUM ALLOYS WITH HELIUM*

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ABSTRACT

A modified tritium trick technique was used to implant three different levels of ^{3}He in V-15Cr-5Ti (wt %) and Vanstar-7 specimens before irradiation in the Fast Flux Test Facility (FFTF). The modifications include: (1) wrapping of the specimens with tantalum foil to minimize oxygen contamination, and (2) a 400°C decay-time treatment to prevent vanadium tritide formation and to produce a ^{3}He bubble distribution similar to that produced during elevated temperature irradiation. Preliminary results show that both modifications were successful. However, the tritium removal step at 700°C was probably too excessive, especially at higher helium levels, because large ^{3}He bubbles formed in the grain boundaries and severely embrittled the V-15Cr-5Ti alloy. Reduction of the tritium removal step to 400°C should alleviate this problem. Vanstar-7 specimens consistently absorbed about half as much tritium, and subsequently contained half as much ^{3}He as V-15Cr-5Ti. Implanting ^{3}He in vanadium alloys via the tritium trick offers a convenient technique to study the mechanism of helium embrittlement without irradiation and should provide a rapid screening method to help develop embrittlement-resistant vanadium alloys.

KEY WORDS: vanadium alloys, V-15Cr-5Ti, Vanstar-7, tritium trick, helium implantation, helium bubbles, helium embrittlement

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DISCLAIMER

1. INTRODUCTION

In developing a structural material for a fusion reactor first wall, it is very desirable to test specimens under conditions where helium is created to the levels expected with 14-MeV neutrons. Since this is not possible in present fission reactors with vanadium alloys, it has been necessary to add helium to the material before irradiation. An undesirable consequence of this sort of simulation is that the swelling behavior and effects on mechanical properties may be different from those that will be encountered with 14-MeV neutron irradiations. Therefore, one must be very careful in interpreting postirradiation tensile tests on materials that are preimplanted with helium levels that would take several years to produce under actual conditions. One method of preimplanting helium is the so-called "tritium trick" where specimens are saturated with tritium which is allowed to decay to ^3He. The tritium trick was selected as the method of implanting vanadium alloys for the Materials Open Test Assembly (MOTA) experiments in the Fast Flux Test Facility (FFTF). The tritium trick technique has the advantages of being able to charge large numbers of samples with high concentrations of ^3He in a relatively short time, and accommodating thick specimens that yield mechanical properties data [1]. An excellent description of the tritium trick has been presented by Remark et al. [2]. A disadvantage of the technique is that under neutron irradiation a percentage of the ^3He is converted back to tritium which can diffuse out of the specimen.

The tritium trick procedure described by Remark et al. [2] was modified to meet our desired simulation requirements for the vanadium

alloys being investigated in the MOTA experiments. In our procedure, the hold-time or decay period was conducted at 400°C instead of room temperature to prevent the formation of vanadium tritides. Phase studies by Schober [3] show that vanadium tritides should not be stable above ~200°C. Another reason for conducting the decay process at 400°C was the belief that the resultant ^{3}H distribution in the microstructure would better simulate those expected in a fusion reactor first wall at elevated temperatures. Since decay times as long as two or three weeks at 400°C were needed, the vanadium alloy specimens were wrapped in tantalum foil to help prevent oxygen contamination. It is well known that oxygen can seriously reduce the ductility of vanadium alloys [4]. Therefore, it was hoped that the tantalum foil would getter any oxygen in the tritium charging chamber, but not overly restrict the flow of tritium to the specimens. This paper presents the preliminary results of initial tritium charging runs on V-15Cr-5Ti (wt %) and Vanstar-7 [V-10Cr-3Fe-1Zr (wt %)]. The effects of the 400°C decay period and tantalum-wrapping were evaluated as well as the ^{3}He distribution in the metal. Several tests were also performed to determine the effects of ^{3}He on the unirradiated mechanical properties.

2. EXPERIMENTAL PROCEDURE

A schematic diagram of the stainless steel system used to charge the vanadium alloys with tritium is shown in Fig. 1. Tritium is supplied initially as a gas as represented by the container on the extreme left. Tritium gas and impurities are transferred to a bed of activated uranium (tritium purifier) at room temperature. The tritium

is absorbed and converted to uranium tritide (UT_3) while the principle impurity, ^3He, is pumped through the bed and exhausted by the roughing pump. Trace impurities of oxygen, nitrogen, and water vapor are also collected by the purifier. Purified tritium is then readily desorbed from the uranium bed when heated to 400°C, and transferred to a storage vessel of activated uranium. The tritium is stored in this form until it is required in the tritium trick chamber. To release the tritium, the storage vessel is simply heated by a furnace to ~400°C which causes the uranium tritide to decompose. Whenever tritium is transferred from one part of the system to another, the connecting lines are first evacuated by the mechanical pump shown on the right side of the diagram. The stainless steel chamber can be evacuated to low pressures ($\sim 10^{-6}$Pa) by utilizing a 100 l/s ion pump. When tritium is transferred into the chamber, higher pressures are used and they are monitored with a gauge shown in the center of the diagram. The chamber is surrounded by an electrical resistance furnace which can be used to heat specimens to 900°C if necessary. Once the tritium trick procedure has been completed, most of the tritium is pumped back into the tritium storage vessel where it is absorbed and converted back to uranium tritide. (The "getter" action of the uranium bed will typically achieve a vacuum of ~133 Pa.)

The actual procedure used to implant ^3He in two different vanadium alloys, V-15Cr-5Ti and Vanstar-7 will now be described. The chemical compositions of both alloys, determined by the vendor, are given in Table 1. Disks (3-mm diameter) and small sheet tensile specimens (gauge section = 7.6 mm long × 1.5 mm wide × 0.84 mm thick) were first cleaned

ultrasonically in methanol and then wrapped in three separate layers of tantalum foil (0.05-mm thick) to minimize oxygen contamination. The wrapped specimens were placed in the tritium chamber and the chamber pumped down to less than 0.5 Pa with the mechanical pump. This step is represented by the first block of the flowchart shown in Fig. 2. Next, the specimens were heated to 400°C for 16 h under a vacuum (~10^{-6}Pa) provided by ion pumping. This pretreatment, i.e., heating under good vacuum, has been shown to influence the metal surfaces such that tritium take-up is much much more rapid. The tritium was inserted at a pressure of 80 kPa (600 mm Hg) while the specimens were still at 400°C. As the tritium was absorbed by the vanadium alloy specimens (and also the tantalum foil), the chamber pressure dropped. More tritium was bled in to raise the pressure above 53 kPa (400 mm Hg) and it was also absorbed. After several chargings of tritium the system was controlled so that an equilibrium pressure of 53 kPa was established. Under these conditions, the atomic fraction of hydrogen in pure vanadium is ~0.13 [5]. The detailed results of this charging step will be presented in the results section of this paper. The specimens were held at the 53 kPa pressure and 400°C to allow the absorbed tritium to decay to ^{3}He according to the decay reaction, ${}_1{}^3\mathrm{H} \xrightarrow{\beta^-} {}_2{}^3\mathrm{He}$. The half-life for tritium decay is 12.26 years which yields ~1/2% decay per month [2]. In this experiment, specimens were held for 20, 120, and 400 h, to produce three different ^{3}He levels in the alloys. At the end of the decay period, tritium in the chamber was removed by opening the valve to the storage vessel and allowing the tritium to be reabsorbed on the "cold" uranium. Tritium in the vanadium alloys which had not decayed to ^{3}He was removed by

increasing the chamber temperature to 700°C and opening the valve to the ion pump to reduce the chamber pressure to ~10^{-6} Pa. This tritium removal phase lasted 16 h. After cooling the chamber to room temperature, the vanadium alloy specimens were removed and cleaned for ~15 s in an ultrasonically-agitated bath of 1 part concentrated HNO_3, 1 part HF, and 1 part H_2O, by volume. The specimens were rinsed in H_2O and ethyl alcohol, and finally dried in air. This cleaning procedure removed the bulk of tritium surface contamination without pitting the samples.

The small tensile specimens were tested to failure at 600°C under vacuum (<10^{-4} Pa) using a crosshead speed of 8.5×10^{-3} mm/s (0.02 in./min).

The 3-mm diameter disks were prepared for examination by transmission electron microscopy by electropolishing in a Struers Tenupol using a solution of 1 part H_3SO_4 to 7 parts H_2O (by volume) at a temperature of −30°C. Fractographs of selected tensile-tested specimens were made using a scanning electron microscope operating at 30 kV.

3. RESULTS

A. Tritium Take-up

The rate at which tritium is absorbed by the vanadium alloys depends on temperature, pressure, alloy composition, and specimen surface condition. A typical tritium take-up curve at 400°C, as represented by pressure drop as a function of time, is shown in Fig. 3. In this run, all the steps outlined previously were followed and a total 27 grams of V-15Cr-5Ti and Vanstar-7 as well as 12.7 grams of tantalum foil were in the chamber. The pressure dropped quite rapidly from the

initial value of 80 kPa to 27 kPa, at which time the pressure was increased back to 80 kPa. The recharging procedure was repeated as shown until the pressure remained steady at 53 kPa. It should be noted that the takeup curve is not only influenced by the rate of tritium absorption, but also by the mass of the specimens and the chamber volume. The specimens were held at this pressure for a specified time to allow the tritium to decay to ^3He.

B. Residual Tritium Level

After the pump out at 700°C the specimens were removed from the chamber and smears were taken from the surface to measure the tritium activity. Counts of 10^4 disintegrations per second (dps) were not uncommon for some specimens where the tritium was apparently tied up in the surface oxide layer. After acid cleaning in which this layer was removed, the smears usually produced less than 4 dps. Accurate measurement of the residual tritium in the metal itself was obtained by dissolving samples electrolytically in a solution of 1 part H_2SO_4 and 7 parts H_2O, by volume. The gas over the solution was run through a catalytic reactor with oxygen so that any tritium in the gas would be converted to ^3H_2O. Aliquots of the electrolytic solution as well as the ^3H_2O were analyzed by scintillation counting and the residual radioactivity level was determined to be 5×10^{-4} Ci/g. This corresponds to >1 at. ppm tritium in the alloy. Details of this tritium analysis technique will be published elsewhere.

C. ^3He Levels and Distribution

The resultant ^3He levels after using the tritium trick for three different decay times were determined by B. Oliver at Rockwell

International, Canoga Park, California, using a high-sensitivity mass spectrometer system and are presented in Table 2. For the longest hold time of 400 h, an average 303 at. ppm of ^3He was measured in V-15Cr-5Ti, while only 149 at. ppm was found in Vanstar-7. This same ratio of ~2:1, for V-15Cr-5Ti to Vanstar-7, was also obtained for the other two runs with shorter hold times. Good ^3He homogeneity through the thickness of the specimen was demonstrated by dissolving half of one piece of V-15Cr-5Ti (400 h hold time) and analyzing the inner core. As shown in Table 2, the inner core had 295 at. ppm ^3He compared with 303 at. ppm for the entire specimen. The distribution of the ^3He in the microstructure can best be shown by TEM. Figure 4 shows two photomicrographs of V-15Cr-5Ti that contains 303 at. ppm of ^3He. Larger ^3He bubbles were observed in the grain boundaries while very small ^3He bubbles (<2.5 nm in diameter) were in the matrix (Fig. 4a). The small matrix bubbles which are barely visible were situated in small loops that show only residual contrast in this particular micrograph. The loops were created during the process of forming the bubbles. Usually, only one bubble was associated with each loop, but occasionally two or even three were observed. In addition to the grain boundaries, a preferential site for ^3He bubbles was at precipitate matrix interfaces as shown in the photomicrograph in Fig. 4b. Coalescence of the bubbles is evident, especially in the lower portion of the interface. In two separate areas the bubbles have coalesced to form a long continuous bubble or microcrack. There was no evidence of tritide formation in the microstructure.

D. Oxygen Analyses

A new feature of the present tritium trick procedure is that the specimens were wrapped in tantalum foil to prevent gross oxygen contamination while still allowing the tritium to reach the specimens. In order to ascertain whether the foil was effective, oxygen analyses were carried out using a sensitive neutron activation technique. The results of those analyses are present in Table 3. Before the tritium trick was applied, V-15Cr-5Ti had an oxygen level of 560 at. ppm. The uncertainty for this measurement was somewhat less than the others because a much larger sample weight was used. Specimens in batches #1 and #5 were charged with tritium for a period of 400 h and picked up a small amount of oxygen. The level after subsequent tensile testing was essentially unchanged. The small amount of oxygen pick-up should have a small, but measurable effect on the mechanical properties of the material. Based on the results by Gold on the same alloy [ref. 4, p. 298], we estimate that losses in total elongation of 6–8% at room temperature and 2–3% at 600°C might occur at the O_2 levels shown in Table 3.

To further investigate the possible effects of O_2 or other elements picked up during the tritium trick on V-15Cr-5Ti, four tensile specimens were run through the identical procedure used to produce the highest ^3He levels. However, in this experiment, hydrogen was substituted for tritium. Therefore, if no changes in the tensile properties were to occur after annealing for the same times and temperatures in hydrogen, one could confidently assume that all changes due to the tritium trick were due solely to the ^3He. The results of the experiment are given in Table 4. The tensile data in the upper portion of the table are for annealed

V-15Cr-5Ti tested at 25 and 600°C. The data below show the effect of annealing in hydrogen and testing at the same temperatures. Comparison of the two sets of data shows that losses in elongation of several percent occurred after annealing in hydrogen and testing at room temperature. At 600°C, the loss of ductility due to annealing in hydrogen was even less. Since the measured O_2 level in the V-15Cr-5Ti specimen after hydrogen annealing was comparable to those after the tritium trick [see Table 3], we have assumed that the small losses in ductility were caused by the oxygen. Similar loss can be attributed to the oxygen absorbed during the tritium treatment. Any further degradation in ductility in V-15Cr-5Ti after the tritium trick can be associated with the ^3He in the microstructure.

E. Effect of ^3He on Mechanical Properties

Some preliminary work was conducted to determine if the addition of the ^3He has a noticeable effect on either the hardness or tensile properties of V-15Cr-5Ti. The addition of 303 at. ppm ^3He to the alloy (batch #1) apparently increased the microhardness from 239 to 254 DPH. However, the major portion of this increase was actually due to oxygen or other interstitials picked up during the tritium trick procedure while a smaller portion was due to helium bubbles and dislocation loops punched out by the bubbles. This conclusion is based on the hardness value of 250 DPH, measured for V-15Cr-5Ti specimens that were given the same treatment — only using hydrogen instead of tritium. In these latter specimens, helium effects could obviously not have been a factor in the hardness measurements.

The effect of ^3He on the tensile properties of V-15Cr-5Ti at 600°C is shown in Fig. 5. The engineering stress-strain curves for a V-15Cr-5Ti specimen with no helium is given along with curves for those charged with 80 and 303 at. ppm. In unimplanted material, the curve was characterized by a pronounced yield point, a normal initial work hardening range, and then discontinuous or serrated plastic flow until failure occurred. The addition of 80 at. ppm ^3He had little effect on the shape of the curve but produced slight reductions in the uniform and total elongations. The stress-strain curve for the specimen with 303 at. ppm ^3He showed an absence of any yield point behavior, very little elongation (~1%), and no discontinuous plastic flow. The fracture mode for the highest ^3He level was entirely intergranular as shown in the scanning electron micrographs in Fig. 6. Figure 6a was taken at low magnification and shows the entire fracture surface, while Fig. 6b is a higher magnification photomicrograph showing the intergranular nature of fracture.

4. DISCUSSION

The amount of tritium and subsequent ^3He dissolved in pure vanadium and its alloys is dependent on the charging temperature and pressure [5]. The results of this experiment show that the amount of tritium take-up also depends strongly on alloy composition. This was evident in the analyses (Table 2) where the Vanstar-7 alloy consistently had half as much ^3He as V-15Cr-5Ti for all three decay times. However, it is not clear whether the difference in composition affected the solubility of tritium in the alloy or its permeability (by changing the nature of the surface oxide film), or both.

The ^3He was distributed quite uniformly from one area of the specimen to another, as shown by the good agreement in duplicate ^3He values listed in Table 2. Each of the two values shown were taken from different areas of their respective specimens. Reasonable uniformity through the specimen thickness was demonstrated in Table 2 by the analysis where the outer half of the specimen was etched away and the inner core had nearly the same ^3He content (295 at. ppm) as two adjacent areas that were analyzed through the entire thickness (302 and 303 at. ppm). On a microstructural scale, larger numbers and larger sizes of helium bubbles appeared on the grain boundaries and particle/matrix interfaces than in the grain matrices. In fact, extensive coalescence of the bubbles was observed in those boundary regions. Undoubtedly, these vast bubble networks weakened the grain boundaries and were the main reason why the V-15Cr-5Ti tensile specimen with 303 at. ppm ^3He failed intergranularly at low strain. It is not known whether the extensive boundary helium bubble networks were due to bubble coalescence or if larger amounts of ^3He were actually produced in the boundaries by the trapping of tritium. Other investigations have shown that at room temperature, particle/matrix interfaces strongly trap tritium [6]. However, it is not known whether these trapping sites would be effective at 400°C. The trapping of tritium (or hydrogen) in grain boundaries without precipitate particles is also not well understood [7]. More work is needed to show whether trapping of tritium is associated with the growth of large helium bubbles at the grain boundaries.

The method of implanting helium and resultant bubble distribution in the microstructure of vanadium and other structural alloys has a

strong influence on their mechanical properties. Specimens of V-20Ti that were preinjected with ~200 at. ppm helium in a cyclotron at 20°C and then neutron irradiated at 400 to 700°C, had grain boundary bubbles that were only slightly larger than those in the matrix [8]. In subsequent tensile tests, specimens exhibited a decrease in total elongation of only ~20% compared to the unirradiated material. That experiment, and others, where helium is implanted at room temperature, do not provide as good a simulation as those where helium is either injected simultaneously during irradiation or preinjected at elevated temperatures. Schroeder and Batfalsky [9] have shown that austenitic steel specimens had shorter life times and lower ductilities when tested in creep during helium implantation (in-beam) compared to specimens injected at room temperature and subsequently creep-tested at the same temperature. They also found larger helium bubbles in specimens that were implanted at elevated temperatures and observed that bubbles were preferentially located in the grain boundaries. Therefore the choice of using an elevated temperature decay period for the tritium trick is warranted. The problem is that the elevated temperature treatments were excessive, especially for the higher helium levels, and larger bubbles in the grain boundaries caused severe embrittlement before the specimens were irradiated. This difficulty can probably be eliminated by changing the tritium removal procedure. Tritium was removed by annealing at 700°C for 20 hours — a rather potent treatment with regard to bubble growth. Lowering of the removal temperature to 400°C would greatly reduce bubble growth while still permitting tritium removal. Tritium decay and removal would then be conducted at the same temperature.

Moreover, until more is known about the effect of 14 MeV fusion neutrons on vanadium alloys, it is probably a good idea to preimplant helium at the same temperature that the alloy will be irradiated.

From the standpoint of studying the mechanism of helium embrittlement, the present tritium trick procedure with the 700°C tritium removal step and resultant ^3He bubble distribution offer some exciting opportunities. In a matter of weeks, vanadium alloy specimens can be implanted with ^3He and tested — without going into the reactor. Changes in alloy composition and microstructure can be made and tested, such that the alloy development process directed toward alleviating the problem of helium embrittlement will be greatly accelerated. Of course, the final test of any viable alloy would be conducted in the reactor. This concept also has the advantage that, with the low levels of residual tritium in the vanadium alloys, one may work in a well-ventilated contamination zone, instead of a hot cell.

It does not appear that other factors such as oxygen, tritium, or other interstitial contaminants, markedly affected the vanadium alloys. Although some oxygen was picked up during the implantation process, the amounts were so small that only slight elongation losses would be expected in the alloy based on Gold's work [4]. Even more convincing was the experiment run in hydrogen instead of tritium, where the V-15Cr-5Ti specimens showed only some minor losses in ductility. The extent of these losses was actually less that estimated from Gold's data [4] for comparable oxygen levels in V-15Cr-5Ti. Since no specimens were implanted without using protective tantalum foil wraps, there are no data to show how much oxygen would have otherwise been picked up.

However, we encountered a vivid demonstration of the virtue of wrapping with tantalum. During one tritium exposure a brief pressure rise due to a faulty valve occurred. The tantalum foil was found to be discolored and very brittle. But oxygen analysis showed that specimens in this batch actually picked up less oxygen than a subsequent, trouble-free duplicate run. The oxygen pickup would have surely been much greater if tantalum wraps would not have been used. Barring any synergistic effects between helium and interstitial elements, it is therefore felt that the effects of the tritium trick on the ductility of the two vanadium alloys were due mainly to helium.

The initial measurements of several mechanical properties for V-15Cr-5Ti, charged with ^3He, were interesting. A concentration of 303 at. ppm caused only a 7% increase in room temperature hardness but drastically reduced ductility at 600°C. As mentioned earlier, the embrittlement was caused by the rather extensive filling of the grain boundaries with ^3He bubbles. These results generally agree with those of Mattas et al. [10] and Santhanum et al. [11] for the same alloy except that they observed the onset of embrittlement at 700 to 750°C. However, the helium levels used in both previous experiments were about an order of magnitude lower than ours, and more importantly, the helium was implanted at room temperature. As mentioned earlier, this produces a bubble distribution that is different from those produced at elevated temperatures and less embrittlement. Other features of the stress-strain curves (Fig. 5) include the gradual erasure of yield point behavior with increasing ^3He levels and the presence of discontinuous or serrated plastic flow. The disappearance of the yield point behavior

suggests some interaction between dislocations and the helium bubbles, or perhaps, the development of a different mechanism of plastic flow such as grain boundary sliding. Since serrations were present in the stress-strain curves of both ^{3}He-implanted and unimplanted specimens tested at 600°C, it is unlikely that they were caused by ^{3}He. Similar serrations have been observed for stainless steels by Fahr [12] who speculated that such beha may be caused by stress concentrations in the grain boundaries. Further work is needed to identify the mechanisms causing the serrations as well as the yield point behavior.

5. CONCLUSIONS

1. The use of a decay period at 400°C produced a macroscopically uniform ^{3}He distribution throughout the V-15Cr-5Ti and Vanstar-7 alloys without forming vanadium tritide.
2. Microscopically, with 303 at. ppm ^{3}He, large bubbles formed in the grain boundaries that caused V-15Cr-5Ti specimens tested at 600°C to fail intergranularly with little elongation. Only small ductility losses were observed with 80 at. ppm ^{3}He.
3. A more desirable preirradiation microstructure with somewhat smaller grain boundary bubbles can probably be produced by reducing the tritium removal temperature from 700 to 400°C.
4. Oxygen pickup was minimized during the tritium trick by wrapping the specimens in tantalum foil, but, even with this precaution, slight losses in ductility were measured in V-15Cr-5Ti due to the additional oxygen.
5. The Vanstar-7 alloy consistently absorbed about half as much tritium, and subsequently contained half as much ^{3}He, as V-15Cr-5Ti.

6. Implanting ^{3}He in vanadium alloys using the present tritium trick procedure offers a convenient technique to study the mechanism of helium embrittlement without irradiation and should provide a rapid screening method to help develop embrittlement-resistant vanadium alloys.

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FIGURE CAPTIONS

Fig. 1. Schematic diagram of tritium trick apparatus.

Fig. 2. Flowchart of tritium trick procedure.

Fig. 3. Typical tritium-charging curves.

Fig. 4. Microstructure of V-15Cr-5Ti after charging with 303 at. ppm ^{3}He using the tritium trick showing ^{3}He bubbles on (a) grain boundaries, and (b) particle/matrix interfaces.

Fig. 5. Stress-strain curves for V-15Cr-5Ti containing different levels of ^{3}He.

Fig. 6. Fracture surface of V—15Cr—5Ti specimen with 303 at. ppm ^{3}He that was tensile tested at 600°C. (a) Low magnification showing fracture across the entire gauge section, and (b) higher magnification showing that fracture was intergranular.

TABLE 1

CHEMICAL COMPOSITION OF V-15Cr-5Ti AND VANSTAR-7
(WEIGHT PERCENT)

Alloy	Cr	Ti	Fe	Zr	C	O	N
V-15Cr-5Ti	14.5	6.2	—	—	0.032	0.031	0.046
Vanstar-7	9.7	—	3.4	1.3	0.064	0.028	0.052

TABLE 2

ANALYSES OF ^{3}He IN VANADIUM ALLOYS

Alloy	Hold time (h)	Helium Concentration (at. ppm)	
		Measured*	Average
V-15Cr-5Ti	400	303.4 302.2	303
Vanstar-7	400	148.7 149.0	149
V-15Cr-5Ti	120	80.02 80.30	80
Vanstar-7	120	42.33 42.16	42
V-15Cr-5Ti	20	13.52 13.51	14
Vanstar-7	20	6.954 7.035	7
V-15Cr-5Ti (inner core)	400	296.0 293.8	295

*Analyses performed by B. Oliver, Rockwell International, Canoga Park, California.

TABLE 3

ANALYSES OF OXYGEN IN V-15Cr-5Ti

Condition	Oxygen Content (at. ppm)
Before tritium trick	560 ± 150
After tritium trick Batch #1	990 ± 300
After tritium trick Batch #5	1370 ± 300
After tritium trick (Batch #1) and testing at 600°C	1040 ± 300
After tritium trick procedure, using H_2	1030 ± 300

TABLE 4

TENSILE PROPERTIES OF V-15Cr-5Ti — BEFORE AND AFTER ANNEALING IN H_2 AT 400°C

Test Temperature (°C)	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Total Elongation (%)
Solution annealed (annealed 1 h at 1200°C in vacuum)			
25	573	689	26
25	576	693	27
25	562	683	28
600	332	524	20
600	345	546	21
Solution annealed + 400 h in H_2 at 400°C, plus 30 h in vacuum at 700°C			
25	552	662	24
25	600	714	25
600	394	592	19
600	390	579	20

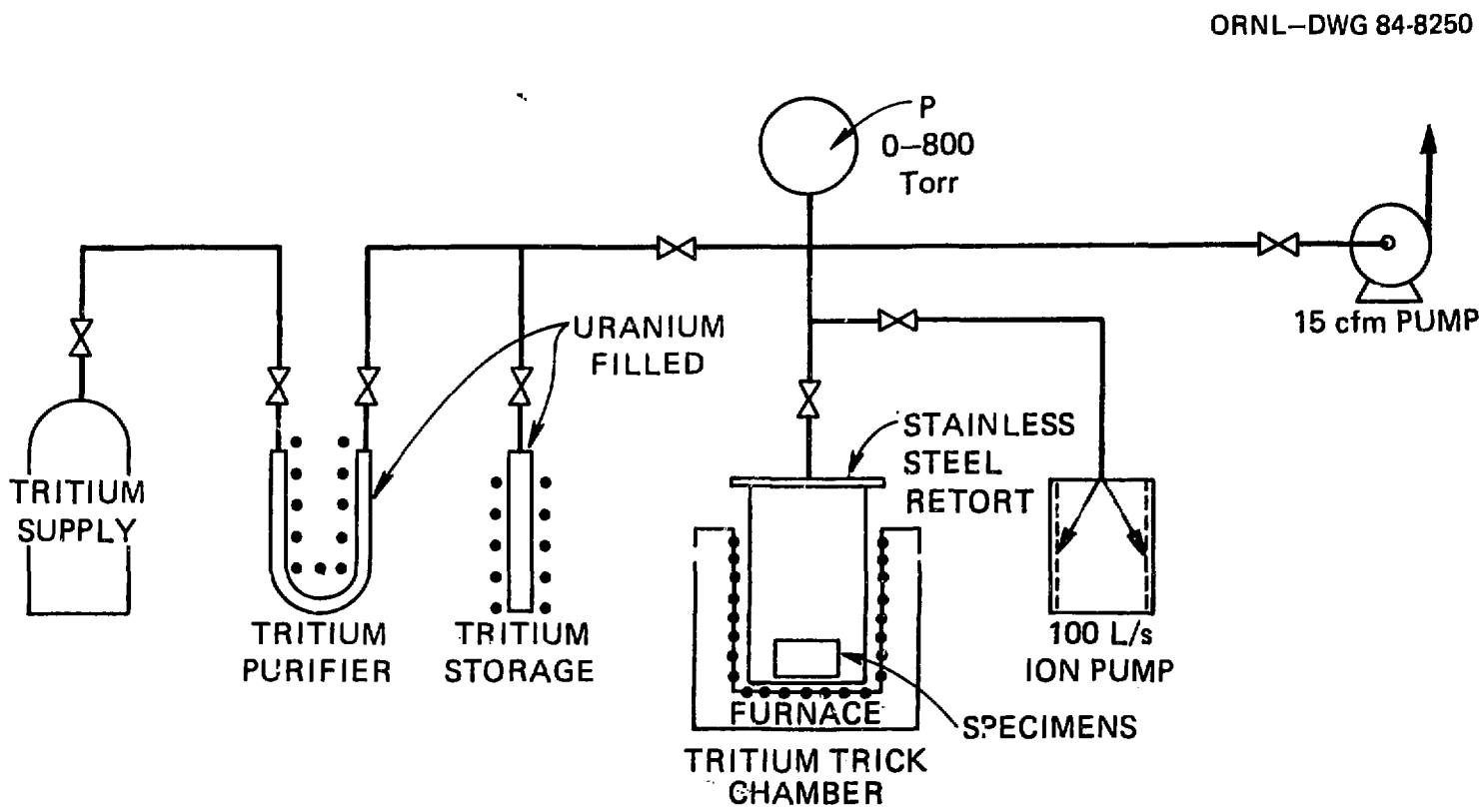

Fig. 1. Schematic diagram of tritium trick apparatus.

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TRITIUM TRICK FLOW CHART FOR VANADIUM ALLOY SPECIMEN

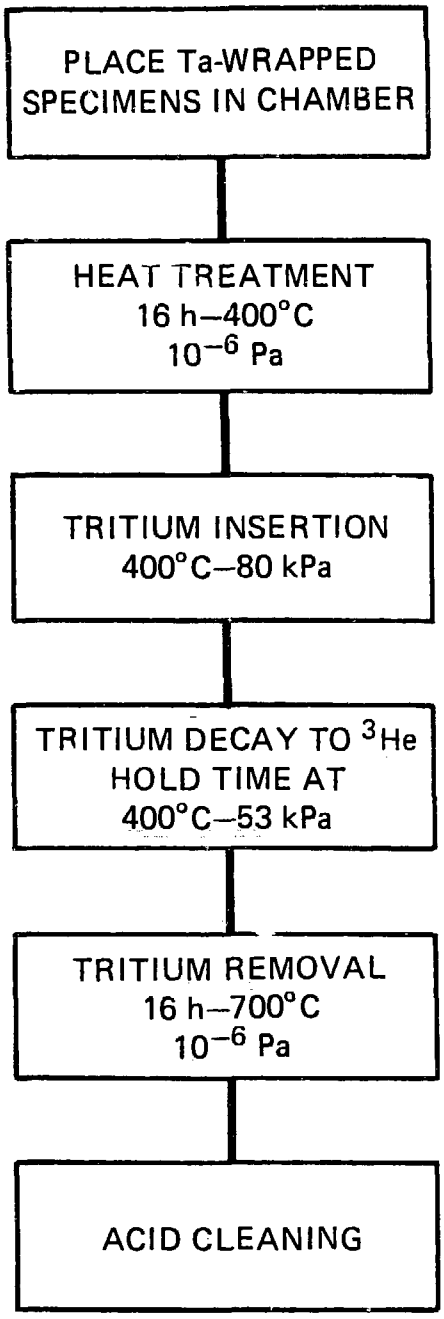


Fig. 2. Flowchart of tritium trick procedure.

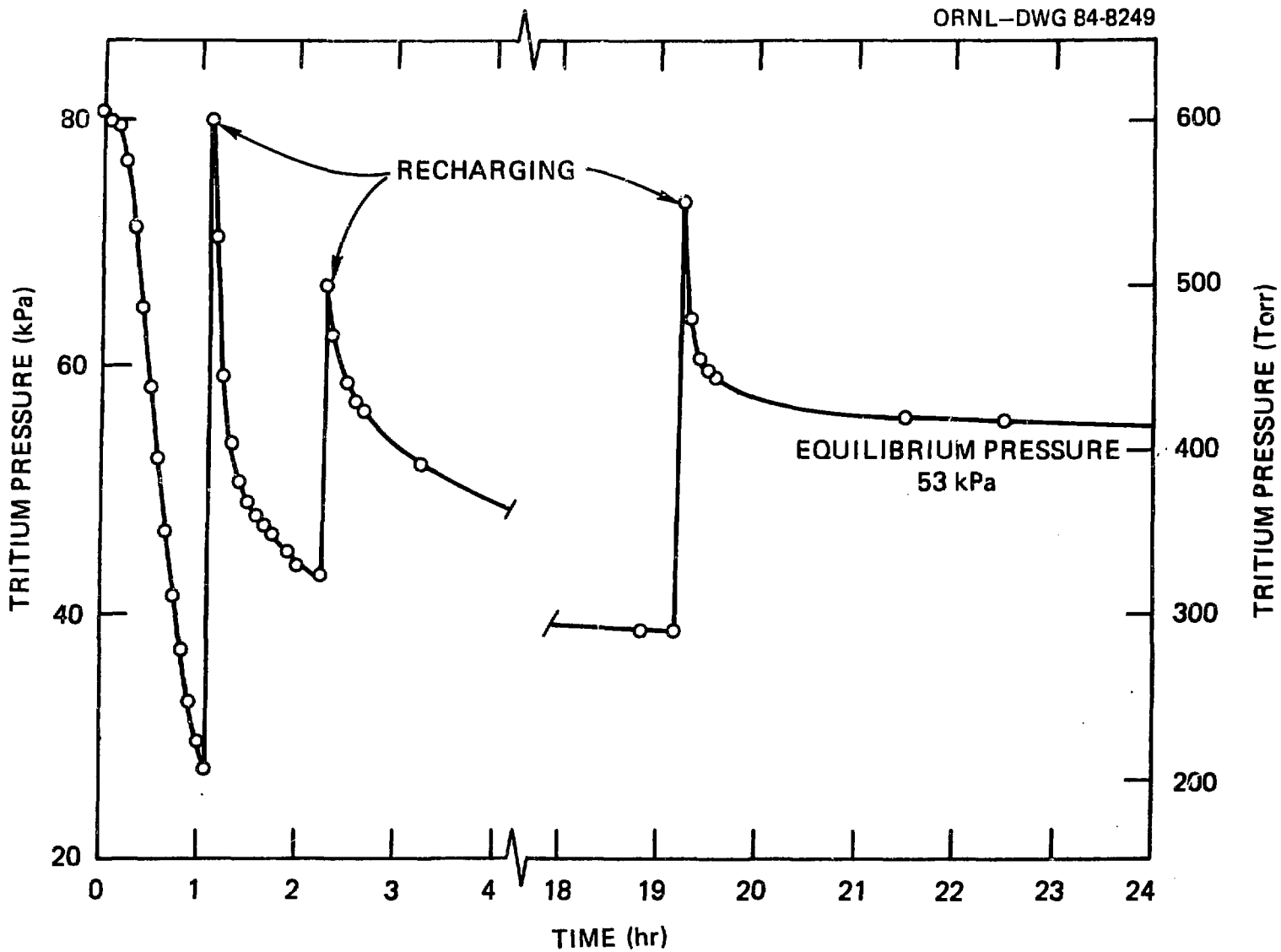


Fig. 3. Typical tritium-charging curves.

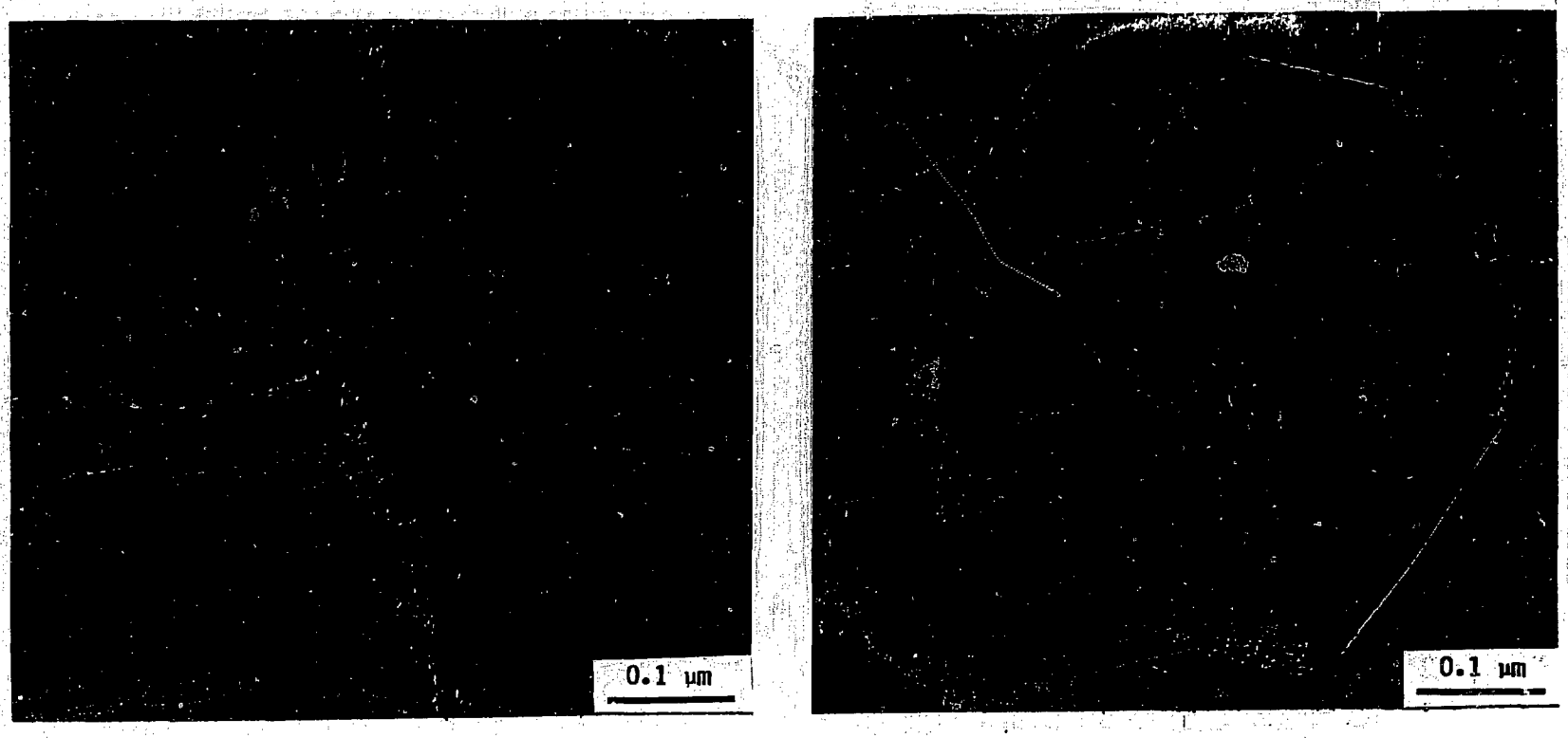


Fig. 4. Microstructure of V-15Cr-5Ti after charging with 303 at. ppm ^{3}He using the tritium trick showing ^{3}He bubbles on (a) grain boundaries, and (b) particle/matrix interfaces.

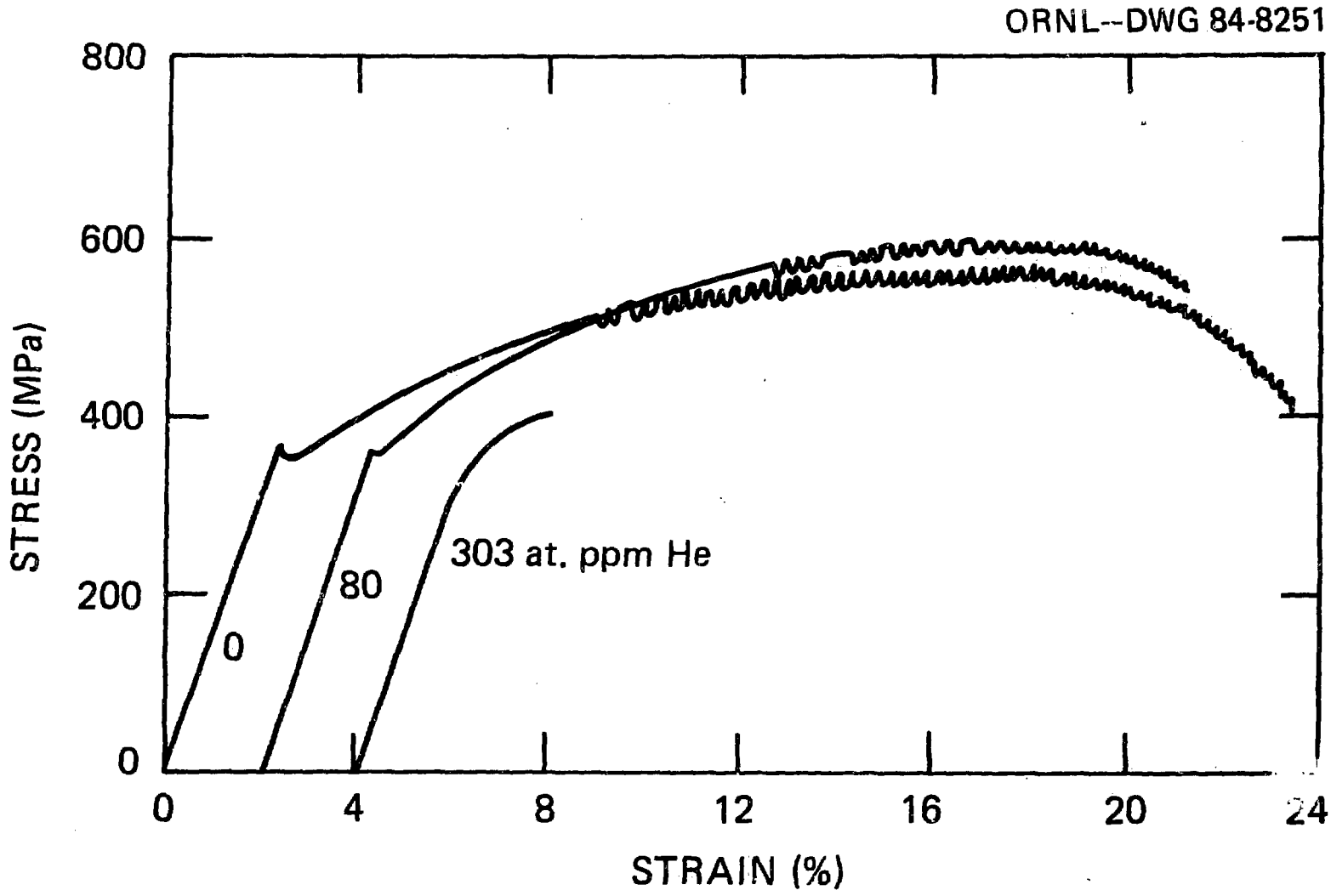
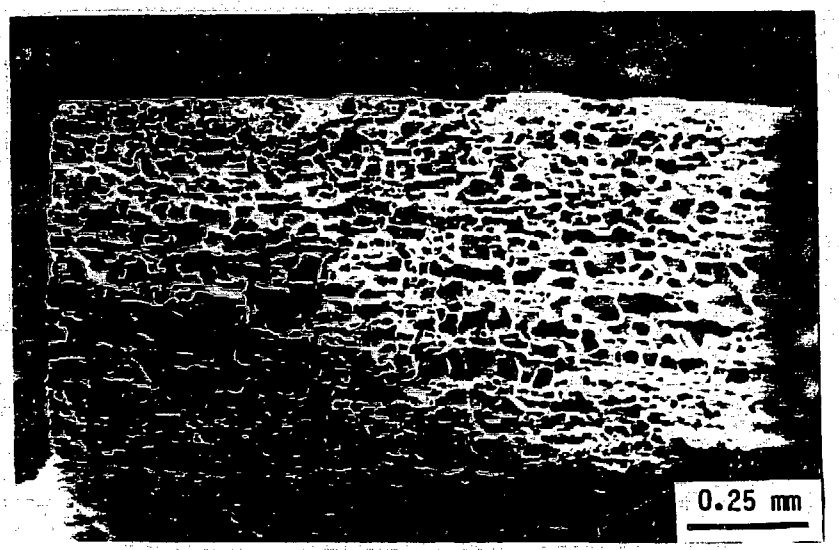
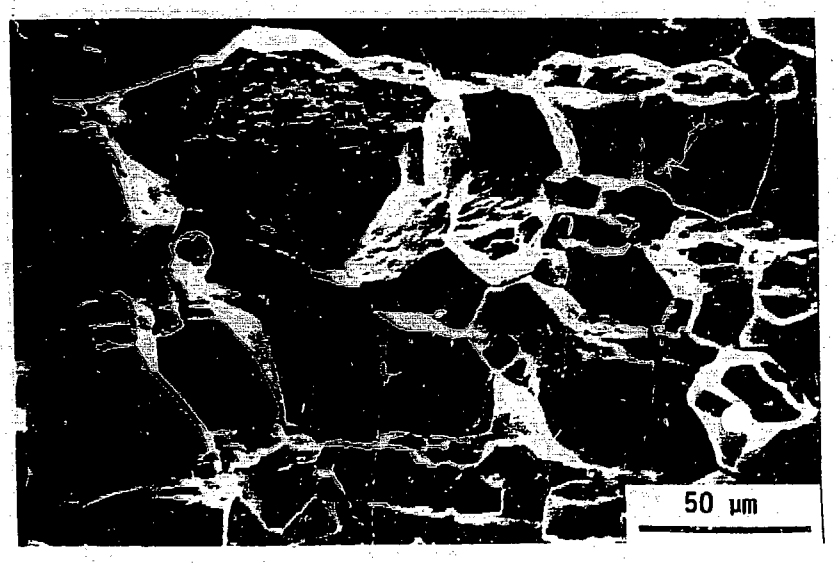


Fig. 5. Stress-strain curves for V-15Cr-5Ti containing different levels of ^{3}He.



(a)



(b)

Fig. 6. Fracture surface of V—15Cr—5Ti specimen with 303 at. ppm ^{3}He that was tensile tested at 600°C. (a) Low magnification showing fracture across the entire gauge section, and (b) higher magnification showing that fracture was intergranular.