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Revision of the Tensile Databae for V-Ti and V-Cr-Ti Alloys Tested at ANL*

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Revision of the Tensile Database for V-Ti and V-Cr-Ti Alloys Tested at ANL*

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Abstract

The published database for the tensile properties of unirradiated and irradiated vanadium-based alloys tested at Argonne National Laboratory (ANL) has been reviewed. The alloys tested are in the ranges of V-(0-18)wt.%Ti and V-(4-15)wt.%Cr-(3-15)wt.%Ti. A consistent methodology, based on ASTM terminology and standards, has been used to reanalyze the unpublished load vs. displacement curves for 162 unirradiated samples and 91 irradiated samples to determine revised values for yield strength (YS), ultimate tensile strength (UTS), uniform elongation (UE) and total elongation (TE). The revised data set contains lower values for UE (-5±2% strain) and TE (-4±2% strain) than previously reported. Revised values for YS and UTS are consistent with the previously-published values in that they are within the scatter usually associated with these properties.

Key Words: Vanadium Alloys and Compounds, Mechanical Properties, Materials Data Base

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1. Introduction

The results of tensile tests performed at Argonne National Laboratory (ANL) on unirradiated and irradiated V-Ti and V-Cr-Ti alloys have been reported in tabular and/or graphical form in the Fusion Reactor Materials Semiannual Progress Reports DOE/ER-0313/2 through DOE/ER-0313/20 [1-9]. Results can also be found in the open literature publications [10,11], but the Progress Reports contain the most detailed set of results for the tensile properties of interest: yield strength (YS), ultimate tensile strength (UTS), uniform elongation (UE) and total elongation (TE). In this current work, the original force-displacement strip chart recordings for these tests have been re-analyzed based on a consistent set of terminology and methods established by ASTM [12]. The motivation for the re-analysis comes from the observation that the reported values of UE and TE appear to be inconsistent with the force-displacement diagrams.

The tensile specimens used in most ANL tensile tests were scaled down from the ASTM dog-bone example [12] to a gauge length of 7.62 mm and a cross-sectional area of ≈ 1 mm² (0.9-1.4 mm²). A uniform crosshead speed of 0.5 mm/min. was used for these tests, giving an effective gauge-length strain rate of 0.11%/s. The samples used to study the effects of oxidation on the tensile properties had nominal gauge length and cross-sectional area of 19 mm and 4.5-mm². Based on crosshead speed, the gauge-length strain rate for these tests was 0.018%/s. Including the oxidation samples, 162 load-displacement curves were re-analyzed for unirradiated vanadium alloys and 91 curves were re-analyzed for FFTF- and HFIR-irradiated alloys.

2. Methodology

The first step in analyzing a force-displacement curve is to determine a linearized slope (k) and zero-displacement point. The methodology used in the current work corresponds most closely to the tangent modulus approach described by ASTM. With most of the loaddisplacement curves for vanadium alloys tested at ANL exhibiting an elongated "S" shape during the initial rise in load, the modulus is determined by the tangent line coinciding with the most number of points in the elongated part of the S-shape. The initial rise in load with crosshead displacement is influenced by the "machine stiffness" and the stiffness of the gauge length. Included in the machine stiffness are all deformations outside the gauge length: load train, grips, pin supports, wide ends of the specimens, local deformation of the specimen at the pin supports, clearance between the hole in the specimen and the pin support, and deformation in the transition region of the specimen. Based on an analyses of 162 force-displacement curves for unirradiated samples, the machine stiffness is $k = 1.20\pm0.20$ kN/mm, corresponding to an effective Young's modulus $E' = 82\pm15$ MPa/%, where the \pm values indicate one standard deviation. Both k and E' are relatively insensitive to temperature. For irradiated samples, $k = 1.76\pm0.34$ kN/mm and E' = 120 ±28 MPa/%. This effective Young's modulus is at least an order of magnitude less than the gauge-length E for vanadium alloys [13]. The observation that k and E' increase with the irradiation-induced hardening of the specimen suggest that the deformation in the non-gauge part plays a significant role in the initial rise in load with crosshead displacement.

For monotonically increasing load vs. displacement curves up to the maximum load, YS is the load (divided by the initial gauge cross-sectional area) corresponding to an offset strain of 0.2%. The offset strain is determined by "analytically" or "graphically" unloading the specimen at a slope corresponding to E'. The intersection of this line with the horizontal axis

determines the offset strain. Some of the load-displacement curves analyzed exhibit discontinuous yielding which consists of a rise to a high load (upper yield point, UYP) followed by a drop in load to a minimum value (lower yield point, LYP) followed by a rise in load with displacement up to the maximum load. For these cases, the 0.2% offset stress may not be a good measure for YS. In the current work, YS is defined to be the minimum of the stress corresponding to 0.2% offset and the stress corresponding to LYP.

UTS is relatively straightforward to determine. For monotonically increasing load up to the maximum load, UTS is simply the maximum load divided by the initial gauge cross-sectional area. In the case of discontinuous yielding, the UYP may represent a higher load than for the continuous part of the curve after the discontinuous yielding. In such cases, the UYP is not to be used in determining UTS. UE is the offset strain corresponding to UTS. While UTS can be uniquely determined, more uncertainty is involved in determining UE, particularly in cases for which the maximum load is nearly constant over a strain range. Although ASTM examples imply that the midpoint of this flat region should be used to determine UE, the maximum offset strain corresponding to the maximum load is used in the current work. The maximum strain at peak load is more characteristic of the uniform elongation of the gauge section prior to necking. The total elongation (TE) corresponds to the offset strain at failure. The same slope used to determine YS and UE is used to determine TE.

Some of the load-displacement curves analyzed exhibited serrations, particularly in the region of the peak load to the failure load. If the stress increased smoothly and continuously with strain between YS and a stress < UTS, followed by serrations, then: the maximum of the serrated portion if the serrations are characterized by an instantaneous drop in stress followed by a more gradual rise (most common case); the average of the serrated portion is used if the serrations are characterized by a more sinusoidal variation with strain; and the minimum of the

serrated portion is used if the serrations are characterized by an instantaneous rise in stress followed by a more gradual decrease.

3. Results

Figure 1 shows an example of a force-displacement curve for a V-4Cr-4Ti (BL-47) sample irradiated at 430°C in FFTF to 27 dpa with 23 appm He and tensile-tested at 425°C. The curve shows the initial change in slope, along with the slight discontinuities with load-cell recording scale change, and the serrations in the region of the UTS. Figure 2 shows the simplifications made in deriving the engineering stress-strain curve from the load-displacement data in Fig. 1. The effective elastic modulus has been used to draw the initial linear portion of the curve and the serrations have been smoothed by taking the minimum values to generate a continuous curve. The results of graphical analysis for YS, UTS, UE and TE are also shown in Fig. 2. The same simplifications have been used in constructing the engineering stress-strain curves for all other cases analyzed.

Figure 3 shows a family of stress-strain curves for V-4Cr-4Ti samples irradiated in FFTF and tested at 500-519°C (BL-47), as compared to an unirradiated sample (BL-71) tested at 500°C. The irradiation-induced hardening and decrease in ductility (UE and TE) are apparent from these curves. The effect is more pronounced for the samples irradiated/tested at 400-430° and less pronounced for samples irradiated and tested at 600°C.

Detailed tabular and graphical results for the revised vs. previously-reported values of YS, UTS, UE and TE are presented in [14]. The results are summarized in the following, with emphasis on the V-4Cr-4Ti alloy.

The difference between revised and previously-reported values for UTS of all alloys tested is insignificant for the majority of the cases analyzed. On the average, the new values differ by -3±15 MPa (-1±3%) for unirradiated alloys and -1±6 MPa (0±1%) for irradiated alloys. Overall, the deviation in UTS is much less than the 10% one would expect from heat-to-heat variation for the same nominal composition alloy. The revised values for YS differ from the previously-reported values by -11±19 MPa (-4±6%) for unirradiated samples and 30±37 MPa (6±7%) for irradiated samples, well within the heat-to-heat variation in YS for structural materials. The differences between revised and previously-reported values of UE and TE are more significant. The previous values are all too high because the displacement due to the non-gauge part of the samples was not properly subtracted from the crosshead displacement. The new values for UE are -5±2% (strain) lower than previously published for both unirradiated and irradiated materials. The new values for TE are -4±2% (strain) lower for both unirradiated and irradiated samples.

Because of the interest in the V-(4-5)Cr-(4-5)Ti alloys, it is worthwhile to establish baseline tensile properties for these alloys as a function of temperature. The particular compositions and heats of interest are: V-3.8Cr-3.9Ti-0.078Si (BL-71, #832665), V-4.1Cr-4.3Ti-0.087Si (BL-47, #9144), V-4.6Cr-5.1Ti-0.031Si (BL-63, #832394) and V-4.9Cr-5.1Ti-0.055Si (BL-72 or T87). Figures 4-7 show the variations of YS, UTS, UE and TE, respectively, with temperature for unirradiated samples. The solid lines represent best-fit cubic equations.

Based on the data presented in Ref. 14, V-4Cr-4Ti exhibits both an increase in strength and a decrease in ductility with irradiation. The degree of hardening and embrittlement decreases with the irradiation/test temperature. Using only results for samples irradiated and

tested at about the same temperature, the increase in UTS is: 79% for 400-430°C, 35% for 500-520°C, and 8% for 600°C. The same pattern holds true for YS but the increases are larger: 162% for 400-430°C, 112% for 500-520°C, and 45% for 600°C. UE decreases with irradiation to strain values of $1.6\pm1.0\%$ for 400-430°C, $3.6\pm1.3\%$ for 500-520°C and $6.5\pm1.1\%$ for 600°C. Similarly, TE decreases to strain values of $5.5\pm1.3\%$ for 400-430°C, $10\pm1\%$ for 500-520°C, and $13\pm2\%$ for 600°C.

3. Discussion

The tensile data for unirradiated and irradiated vanadium alloys tested at ANL have been reviewed and re-analyzed in accordance with ASTM procedures. The resulting values for ultimate tensile strength are in good agreement with values reported previously. On the average, the changes in yield strength values are small and well within the heat-to-heat scatter for this parameter. The differences between the new and old values for YS occur primarily due to different methodologies used in determining YS. In the current work, YS is determined to be the minimum of the 0.2% offset stress and the stress corresponding to the lower yield point. The previous YS values for unirradiated materials were mainly determined from the 0.5% offset stress criteria. For irradiated materials, several different methodologies (proportional elastic limit, upper yield point, 0.2% offset stress, etc.) were used. There is no single "best" way to determine YS for all structural materials and all sample sizes. The main contribution of the current set of values is that they have been determined by a consistent methodology.

The current values for uniform and total elongation are smaller than those reported previously. This arises from properly subtracting non-gauge-length deformation from the total crosshead deformation. For unirradiated and irradiated alloys, the decreases in UE and TE are $5\pm2\%$ and $4\pm2\%$, respectively, where the \pm value refers to one standard deviation. There is a

significant impact on the ductility of the alloys irradiated and tested at 400-430°C. For unirradiated alloys and alloys irradiated/tested at ≥ 500 °C, the revised values for UE are still within the ductile range (as defined by standard design codes).

The tensile data set presented in this current work should be combined with tensile data for vanadium alloys irradiated in other reactors (e.g., EBR-II, HFIR, ATR, BOR-60, HFBR, etc.) at a range of temperatures and neutron damage levels to form a more complete picture of the temperatures, neutron damage levels and helium levels for which the uniform elongation decreases to a low value (e.g. < 2%). Currently, the number of data points for any one alloy is insufficient to determine uniform elongation transition temperatures.

4. Conclusion

Review of the tensile data for V-Ti and V-Cr-Ti alloys tested at ANL indicates that there was a systematic error in the determination of uniform (UE) and total (TE) elongation. The revised values for UE are -5±2% (strain) lower than previously-reported values. The revised values for TE are -4±2% (strain) lower. Of the two, the decrease in UE is more significant, particularly for alloys irradiated/tested at 400-430°C where UE drops below 2% for some of the alloys. More tensile data on irradiated samples are needed in the temperature range of 400±50°C and up to higher fluences to determine the temperature/neutron-damage regimes for which UE decreases to below 2% for the V-Cr-Ti alloys. Other properties (e.g., fracture toughness, Charpy energy, etc.) should be obtained in these same temperature/fluence regimes and factored into the performance analysis to determine transition temperatures/fluences for ductile to brittle behavior of vanadium alloys. Optimization of the Cr, Ti and Si constituents, along with interstitial O, C, and N and other impurities may be called for to achieve the best performance in terms of strength and ductility of vanadium alloys.

References

- 1. B. A. Loomis, R. H. Lee and D. L. Smith, in Fusion Reactor Materials Semiannual Progress Report for Period Ending March 31, 1987, DOE/ER-0313/2, US Department of Energy, Office of Fusion Energy, pp. 250-258.
- 2. B. A. Loomis, R. H. Lee and D. L. Smith, in Fusion Reactor Materials Semiannual Progress Report for Period Ending September 30, 1987, DOE/ER-0313/3, US Department of Energy, Office of Fusion Energy, pp. 246-253.
- 3. B. A. Loomis, L. J. Nowicki and D. L. Smith, in Fusion Reactor Materials Semiannual Progress Report for Period Ending March 31, 1995, DOE/ER-0313/18, US Department of Energy, Office of Fusion Energy, pp. 265-272.
- 4. H. M. Chung, L. Nowicki, D. Busch and D. L. Smith, in Fusion Reactor Materials Semiannual Progress Report for Period Ending December 31, 1995, DOE/ER-0313/19, US Department of Energy, Office of Fusion Energy, pp. 17-21.
- 5. B. A. Loomis, L. J. Nowicki, and D. L. Smith, in Fusion Reactor Materials Semiannual Progress Report for Period Ending September 30, 1993, DOE/ER-0313/15, US Department of Energy, Office of Fusion Energy, pp. 219-221.
- 6. H. M. Chung, B. A. Loomis, L. Nowicki and D. L. Smith, in Fusion Reactor Materials Semiannual Progress Report for Period Ending December 31, 1995, DOE/ER-0313/19, US Department of Energy, Office of Fusion Energy, pp. 77-82.

- 7. H. M. Chung, L. Nowicki and D. L. Smith, in Fusion Reactor Materials Semiannual Progress Report for Period Ending June 30, 1996, DOE/ER-0313/20, US Department of Energy, Office of Fusion Energy, pp. 78-83.
- 8. H. M. Chung, L. Nowicki and D. L. Smith, in Fusion Reactor Materials Semiannual Progress Report for Period Ending June 30, 1996, DOE/ER-0313/20, US Department of Energy, Office of Fusion Energy, pp. 84-86.
- 9. K. Natesan and W. K. Soppet, in Fusion Reactor Materials Semiannual Progress Report for Period Ending December 31, 1995, DOE/ER-0313/19, US Department of Energy, Office of Fusion Energy, pp. 50-53.
- B. A. Loomis, R. H. Lee, D. L. Smith and J. R. Peterson, J. Nucl. Mater. 155-157
 (1988) 631-638.
- 11. H. M. Chung, B. A. Loomis and D. L. Smith, J. Nucl. Mater. 233-237 (1996) 466-475.
- 12. 1996 Annual Book of ASTM Standards, Section 3: Metals Test Methods and Analytical Procedures, Volume 03.01: Metals -- Mechanical Testing; Elevated and Low-Temperature Tests; Metallography; Designation E 6 (Standard Terminology Relating to Methods of Mechanical Testing, pp 17-26) and Designation E 8 (Standard Test Methods for Tension Testing of Metallic Materials, pp. 55-96), ASTM, West Conshohocken, PA.
- 13. W. A. Simpson, in Fusion Reactor Materials Semiannual Progress Report for Period Ending March 31, 1994, DOE/ER-0313/16, US Department of Energy, Office of Fusion Energy, pp. 258-259.

14. M. C. Billone, "Revised ANL-Reported Tensile Data for Unirradiated and Irradiated (FFTF, HFIR) V-Ti and V-Cr-Ti Alloys," to be published in Fusion Reactor Materials Semiannual Progress Report for Period Ending December 31, 1997, DOE/ER-0313/23, US Department of Energy, Office of Fusion Energy.

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