Effect Of Low Temperature Irradiation In ATR On The Mechanical Properties Of Ternary V-Cr-Ti Alloys - M. L. Hamilton, M. B. Toloczko, B. M. Oliver and F. A. Garner (Pacific Northwest National Laboratory)*

OBJECTIVE

This experiment was designed to allow the relationship between shear punch and tensile data to be established for vanadium alloys irradiated at low temperatures in the ATR-A1 experiment.

SUMMARY

Tensile tests and shear punch tests were performed on a variety of vanadium alloys that were irradiated in the Advanced Test Reactor (ATR) at temperatures between 200 and 300°C to doses between 3 and 5 dpa. Tests were performed at room temperature and the irradiation temperature. The results of both the tensile tests and the shear punch tests show that following low temperature irradiation, the yield strength increased by a factor of 3-4 while the ultimate strength increased by a factor of approximately 3. Uniform elongation and tensile reduction in area show that the ductility diminishes following irradiation. The correlation between uniaxial ultimate strength and effective shear maximum strength was in excellent agreement with previous studies on other materials. Using the room temperature test data, the correlation between uniaxial yield strength and effective shear yield strength was in excellent agreement with previous studies on other materials. The yield strength data obtained at the irradiation temperature did not fit the room temperature correlation.

PROGRESS AND STATUS

Introduction

Vanadium-base alloys are considered to be attractive candidates for low activation structural materials in future fusion power devices. Most attention is currently focused on V-Cr-Ti alloys because of their relatively attractive mechanical properties after irradiation at relatively high temperature and because these alloys are resistant to void swelling. [1] Recent studies at lower temperatures, however, have shown significant degradation in mechanical properties during radiation, with large increases in strength and large decreases in ductility observed following irradiation at temperatures below 400°C. [2] The purpose of the current work is to provide further data on the change in mechanical properties of V-Cr-Ti alloys following low temperature irradiation. Mechanical properties were determined from tensile tests and shear punch tests; the latter is a TEM disk-based test developed to provide strength and ductility information. [3-9] This experiment is the first in which the tensile-shear punch correlation work has been extended to a bcc material, and it is the first time that the viability of the correlation has been evaluated at elevated test temperatures.

Experimental Procedure

The alloys of interest were V-3Cr-3Ti, V-4Cr-4Ti, V-5Cr-5Ti and V-6Cr-3Ti. These alloys will be referred to as V33, V44, V55, and V63, respectively. All were annealed at 1000°C prior to the irradiation for either one hour (V44 and V55) or two hours (V33 and V63). These

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alloys were irradiated as both tensile and TEM specimens in the ATR mixed spectrum reactor in the A1 experiment at nominal temperatures of 200 and 300°C to doses between 3 and 5 dpa. [10] The specimens were irradiated in lithium-bonded subcapsules that were shielded to reduce the number of thermal neutrons. The details of the temperature and dose history for each alloy and specimen type are given in Table 1. As shown in Table 1, tensile specimens of V63 were not available for irradiation.

Table 1. Temperature and dose history of each combination of alloy and specimen geometry.

Alloy	Tensile Specimens		TEM Specimens	
	Actual Irradiation Temp. (°C)	Dose (dpa)	Actual Irradiation Temp. (°C)	Dose (dpa)
V33	205	3	205	3
	295	3	293	4.7
V44	229	3.5	205	3
	293	4.7	293	4.7
V55	229	3.5	205	3
	293	4.7	293	4.7
V63			205	3
			293	4.7

The tensile specimens, cut from sheet stock by electro-discharge machining (EDM), were S1 miniature specimens with nominal gauge dimensions of 5 mm x 1.2 mm x 0.25 mm (0.2 in. x 0.05 in. x 0.010 in.). Specimen thickness ranged from 0.25 to 0.36 mm (0.010 to 0.014 in.) between the alloys , but for any one alloy, the specimen thickness was uniform across the specimen to within 0.01 mm (0.0005 in.). Tensile tests were performed at a crosshead speed of 0.127 mm/min (0.005 in./min), yielding an initial strain rate of 4×10^{-4} sec^{-1}. For each irradiation condition, tests were performed at the actual irradiation temperature and at room temperature. Room temperature tests were performed in air. Specimens tested at elevated temperature required about 90 minutes for heatup and stabilization prior to testing in static argon. One test was performed for each alloy/irradiation condition.

Reduction of area (RA) measurements were obtained using SEM photomicrographs taken from each of the tensile specimen fracture surfaces. RA measurements were obtained on both irradiated and unirradiated test specimens using calibrated digital image analysis software.

Shear punch tests were performed using standard 3 mm diameter TEM disks of the same thickness as the tensile specimens. TEM disks were also cut from sheet stock by the EDM method. Nominal specimen thickness varied from alloy-to-alloy, but for any one alloy, the specimen thickness was uniform across the TEM disk to within 0.005 mm (0.0002 in). Tests were performed at a crosshead speed of 0.127 mm/min (0.005 in./min). Specimen displacement during a test was assumed to be equal to crosshead displacement. For each irradiation condition, tests were performed at the actual irradiation temperature and at room temperature. Room temperature tests were performed in air. Specimens tested at elevated temperature required about 90 minutes for heatup and stabilization prior to testing in slowly flowing argon. One test was performed for each alloy/irradiation condition.

Small pieces of several tested TEM specimens were also analyzed for helium and hydrogen content to determine whether there was any effect of gas generation from the irradiation. The helium analyses were conducted using the high-sensitivity isotope dilution analysis system at PNNL. Details of the system have been presented elsewhere. [11] Hydrogen analyses were conducted using a new facility recently developed at PNNL for measurement of hydrogen in irradiated materials. [12]

Results

Tensile data

The tensile results are given in Table 2, and are shown in Figures 1-4 along with other vanadium alloy tensile data that were generated in this same experiment by researchers at ANL and ORNL. [13,14] Significant strengthening was observed in the V33, V44, and V55 alloys following irradiation at both temperatures (recall that tensile specimens of the V63 alloy were not irradiated). Yield strength typically increased by at least a factor of 3-4, and uniform elongation (UE) decreased typically to less than 1%. No significant differences were observed between alloys. As shown in the figures, these results are very consistent with the vanadium tensile data generated by the ANL and ORNL researchers [13, 14].

Reduction of area measurements were obtained since uniform elongation values from the tensile tests were so low. Figure 5 shows that irradiation caused significant changes in RA as well as in UE. RA dropped from over 90% in the unirradiated condition to about 60-70% for most tests after irradiation at ~200°C and to 5-30% for most tests after irradiation at ~300°C. Figures 6a – 6r give the photographs from which the reduction of area measurements were taken. It is clear that the amount of deformation at the fracture has decreased with irradiation; fractures shifted from a chisel line in the unirradiated condition to a surface with some ductility following irradiation at ~200°C and to almost a flat surface following irradiation at ~300°C.

Shear punch data

Effective shear strength data are shown in Figures 7 and 8. The trends exhibited by these data are almost identical to those observed in the tensile data, i.e., significant strengthening occurred at all temperatures for all alloys. The shear punch data also showed the same test temperature-dependent strength changes that are apparent in the tensile data. The effective shear stress data indicate that the V63 alloy behaved similarly to the other three alloys.

Gas analysis data

Helium and hydrogen concentrations were measured in six of the irradiated and tested shear punch specimens and in selected control specimens. The results are shown in Figure 9.

Table 2. Tensile data on vanadium alloy specimens irradiated in ATR-A1.

Alloy	Specimen ID	Dose	Irr. Temp. (C)	Test Temp. (C)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)	RA (%)
V33	P145	0	-	205	126	289	10.8	24.0	97
	P142	0	-	295	102	179	13.5	19.7	97
	P141	3	205	22	987	998	0.5	5.1	68
	P140	3	205	205	813	862	0.4	2.0	27
	P144	3	295	22	989	1007	0.4	2.4	35
	P143	3	295	295	931	931	0.2	1.5	5
V44	P848	0	-	22	349	414	5.9	10.6	91
	P851	0	-	22	327	457	14.5	31.1	93
	P850	0	-	164	271	388	12.9	23.7	95
	P853	3.5	229	22	1062	1101	0.4	5.4	61
	P845	3.5	229	229	888	901	0.1	5.7	64
	P842	4.7	293	22	992	1120	1.2	2.4	18
	P841	4.7	293	293	976	982	0.3	2.0	9
V55	P740	0	-	22	282	448	17.6	31.6	90
	P742	0	-	229	205	322	11.8	21.8	95
	P743	0	-	293	226	369	13.3	22.6	88
	P747	3.5	229	22	1130	1132	0.2	6.1	62
	P753	3.5	229	229	992	994	0.2	6.1	66
	P752	4.7	293	22	1053	1072	0.9	3.1	19
	P751	4.7	293	293	935	943	0.3	5.3	27

In the irradiated samples, measured helium levels ranged from 1.2 to 3.0 appm. Reproducibility between duplicate helium analyses averaged ~2%, somewhat higher than the analysis system reproducibility of ~0.5%, but consistent with previous results that have shown higher levels of helium heterogeneity in materials that have a boron impurity as suggested below. [15]

Measured hydrogen levels in the same irradiated samples ranged from 237 to 480 appm . Reproducibility between the duplicate analyses averaged ~20% and is consistent with the reproducibility observed in standard hydrogen-containing steels. Measured hydrogen levels in the unirradiated alloys showed a higher average variability of ~60%. The reason for the higher variability in the unirradiated specimens is not clear.

Discussion

Gas analyses

Measured helium contents in the irradiated alloys were about a factor of 10 higher than calculated, suggesting the presence of boron at impurity levels. In spite of the large variance from the expected helium level, the few appm He present are not likely to have affected the mechanical properties of the alloys.

Measured hydrogen levels are about two orders of magnitude higher than calculated. The reason for this is not clear, but since approximately the same hydrogen levels were measured in both the unirradiated and irradiated alloys, it appears that much of the hydrogen was present prior to irradiation thus suggesting either that no additional hydrogen was generated during the irradiation, or that any hydrogen generated diffused out of the samples during the irradiation. It is unlikely that the hydrogen present in the materials prior to irradiation would have affected the changes in mechanical properties due to irradiation.

Tensile and shear punch data

The tensile traces on the unirradiated material exhibited both a yield plateau (or evidence of a yield plateau for those tests where the phenomenon was less distinct) and serrated yielding at all test temperatures. The corresponding shear punch traces exhibited neither of these phenomena. However, in tests of other materials such as low carbon steels and aluminum alloys, the fine detail observed in tensile tests, such as a Luders plateau or serrated yielding, has been observed in corresponding shear punch tests. Both tensile and shear punch traces from unirradiated specimens exhibited a large amount of plastic deformation as well. The UTS was typically 40-60% higher than the YS in both types of tests for the unirradiated condition.

The tensile test traces obtained on irradiated specimens exhibited none of the features observed in the traces from the unirradiated specimens. Load increased rapidly to the maximum value with very little work hardening; the UTS was generally within a few percent of the YS. The test traces from irradiated specimens typically exhibited relatively sharp 'peaks' at maximum load and since no serrations or yield plateaus were observed, it appears that the irradiation-induced defects were much more effective than interstitial atmospheres at pinning dislocations. These features suggest that dislocation channeling is the prevalent deformation mechanism in the irradiated alloys, as has indeed been recently confirmed. [16]

While UE was consistently very low, and exhibited no variation with irradiation temperature, reduction of area exhibited a significant difference with irradiation temperature, being consistently much lower after irradiation at about ~300°C. This suggests that although neither YS nor UE varied significantly with irradiation temperature, one might expect that the fracture toughness of these alloys after irradiation at ~300°C would be much lower than following irradiation at ~200°C.

Tensile-shear punch correlation

While the irradiation temperature and dose received by the TEM specimens was often significantly different than that for the corresponding tensile specimens, it was judged worthwhile to construct a correlation between uniaxial and effective shear yield and maximum strengths. The uniaxial yield and the effective shear yield strength data are compared in Figure 10. Two linear regression lines are shown for the yield strength correlation in Figure 10. One is fit to all the data, while the other is fit to just the room temperature data. Both correlations have nearly the same slope, and the slope is in good agreement with previous correlations on other unirradiated and irradiated materials [4, 6-9]. However, as is evident in Figure 10, the tensile and shear punch data obtained from elevated temperature tests do not fall on the same line as the data obtained from room temperature tests. An examination of the both the tensile and the shear punch test traces revealed that the shear punch traces obtained from elevated temperature tests exhibited a very smooth transition from linear elastic to plastic behavior making it difficult to exactly define the yield point. In keeping with the standard method of choosing the yield point as the deviation from linearity, the yield point was taken as the earliest discernable deviation from linearity, which resulted in low values for the effective shear yield stress.

The reason for the smooth transition from linear elastic to plastic behavior is not understood at this time. It has been suggested that an offset effective shear yield stress be used in a manner analogous to the 0.2% offset yield strength. This was investigated, and due to the geometry of the deformation zone of the shear punch test, a 0.2% offset shear strain is equivalent to only a fraction of the width of the trace line. Thus, such an offset would have no significant effect on the measurement of the effective shear yield strength value.

The correlation for maximum strength, as shown in Fig. 11, is in excellent agreement with previous maximum strength correlations obtained from unirradiated and irradiated materials, and as with previous correlation studies, the maximum strength correlation clearly exhibits less scatter than the yield strength correlation [4, 6-9]. Unlike the yield strength correlation, there is no stratification of the values obtained at room temperature and at elevated temperature.

Summary And Conclusions

Tensile and shear punch tests on vanadium alloys irradiated in ATR at ~200-300°C to 3-5 dpa indicate that significant strengthening and loss of ductility are observed after a small amount of low temperature irradiation and that strength at the irradiation temperature is slightly lower than at room temperature. RA is significantly lower following irradiation at ~300°C than at ~200°C. No significant difference was observed between V33, V44, V55 and V63. The tensile-shear punch relationship was as expected for both the yield and the maximum load conditions, but some variability that is not well understood was observed for the yield condition.

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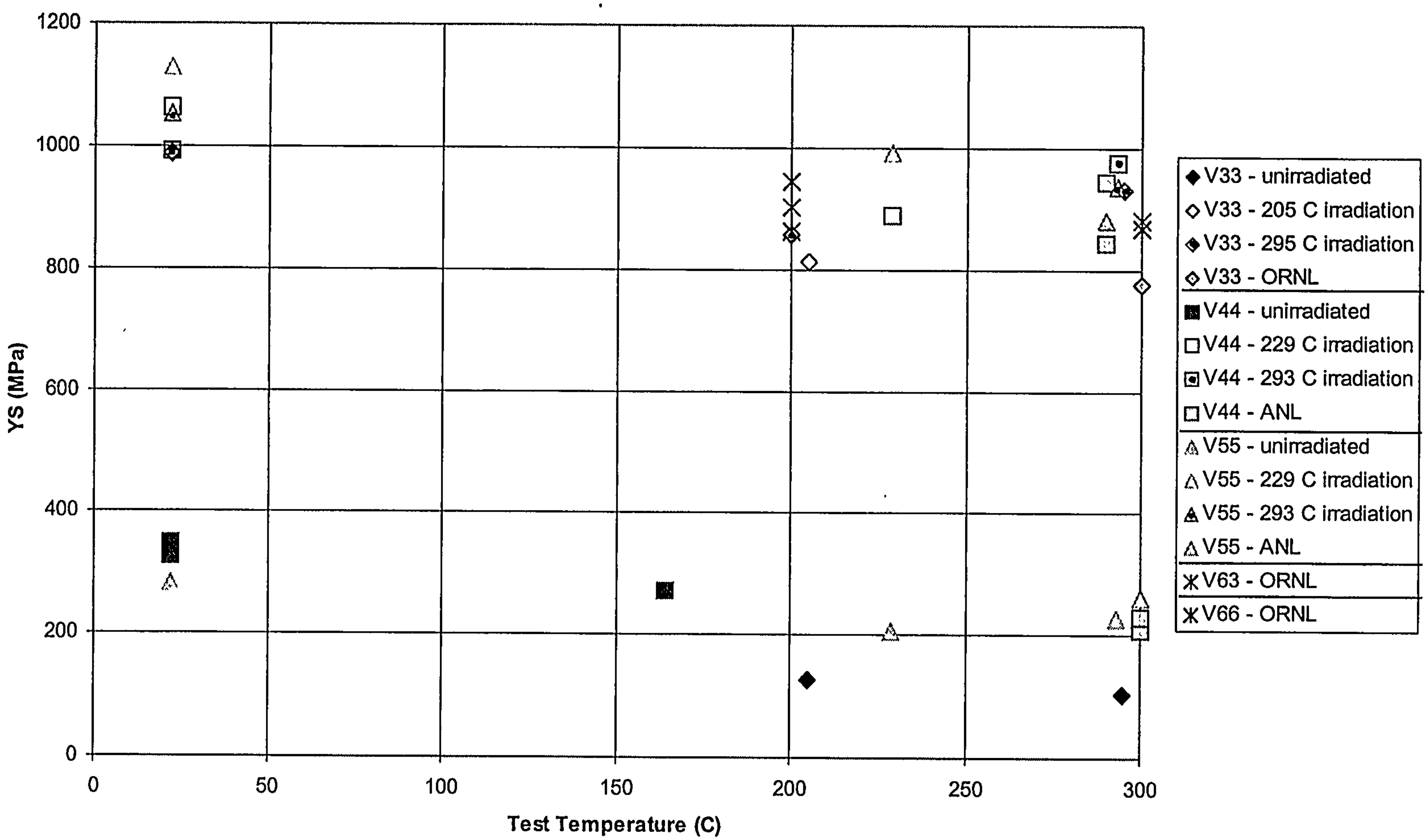

Figure 1 . Yield strength of vanadium alloys irradiated in ATR.

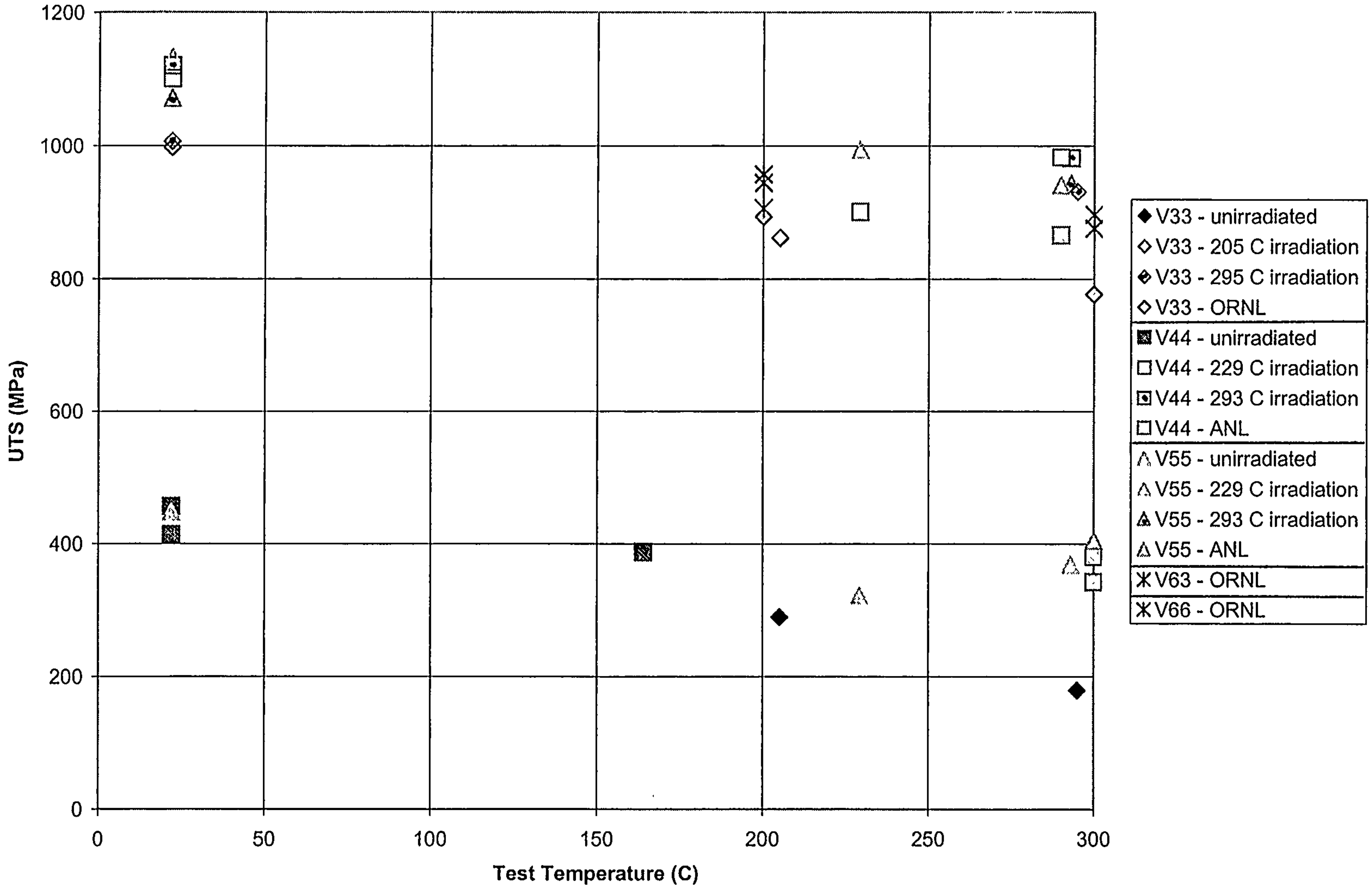

Figure 2. Ultimate strength of vanadium alloys irradiated in ATR.

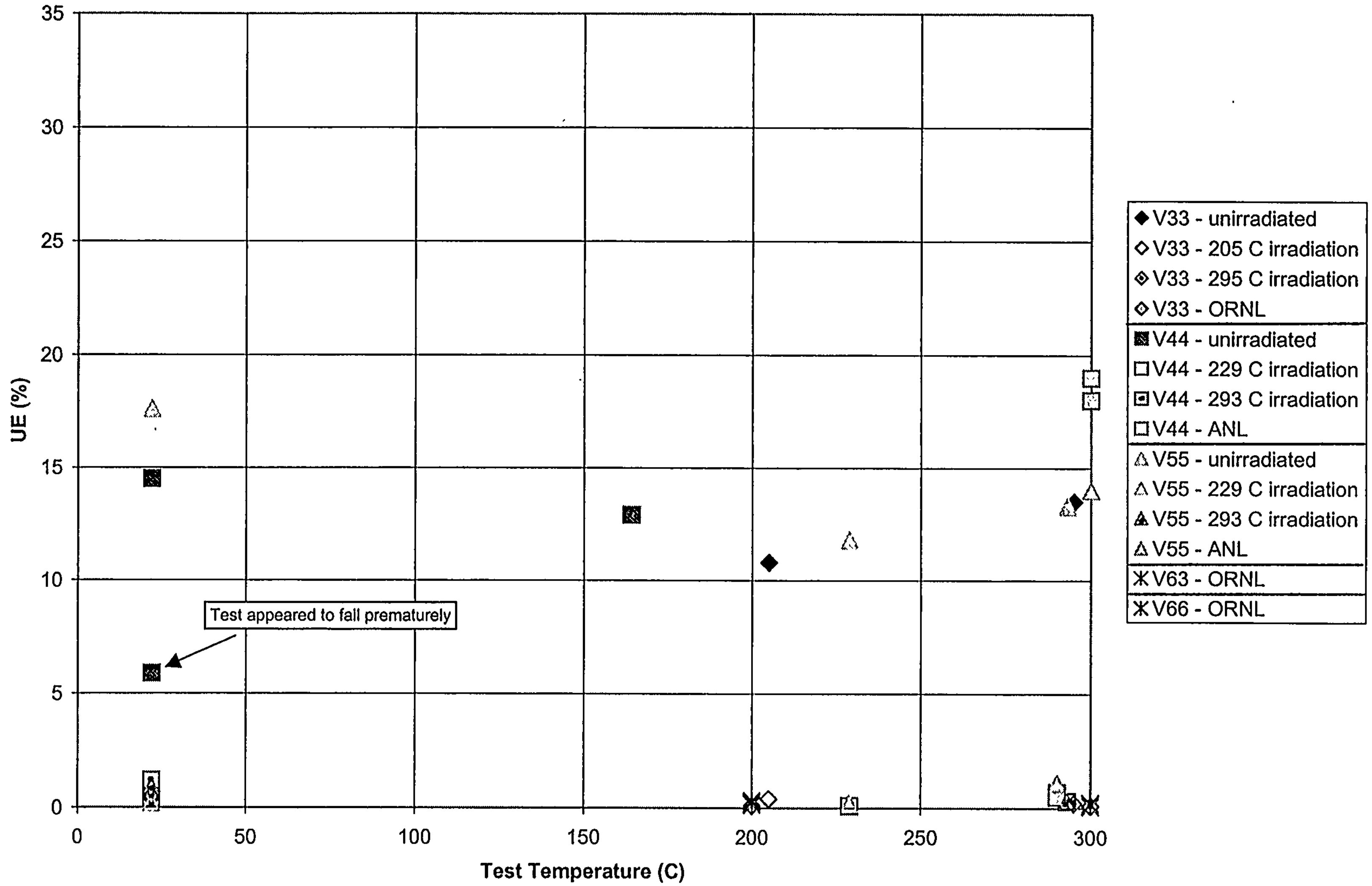

Figure 3. Uniform elongation of vanadium alloys irradiated in ATR.

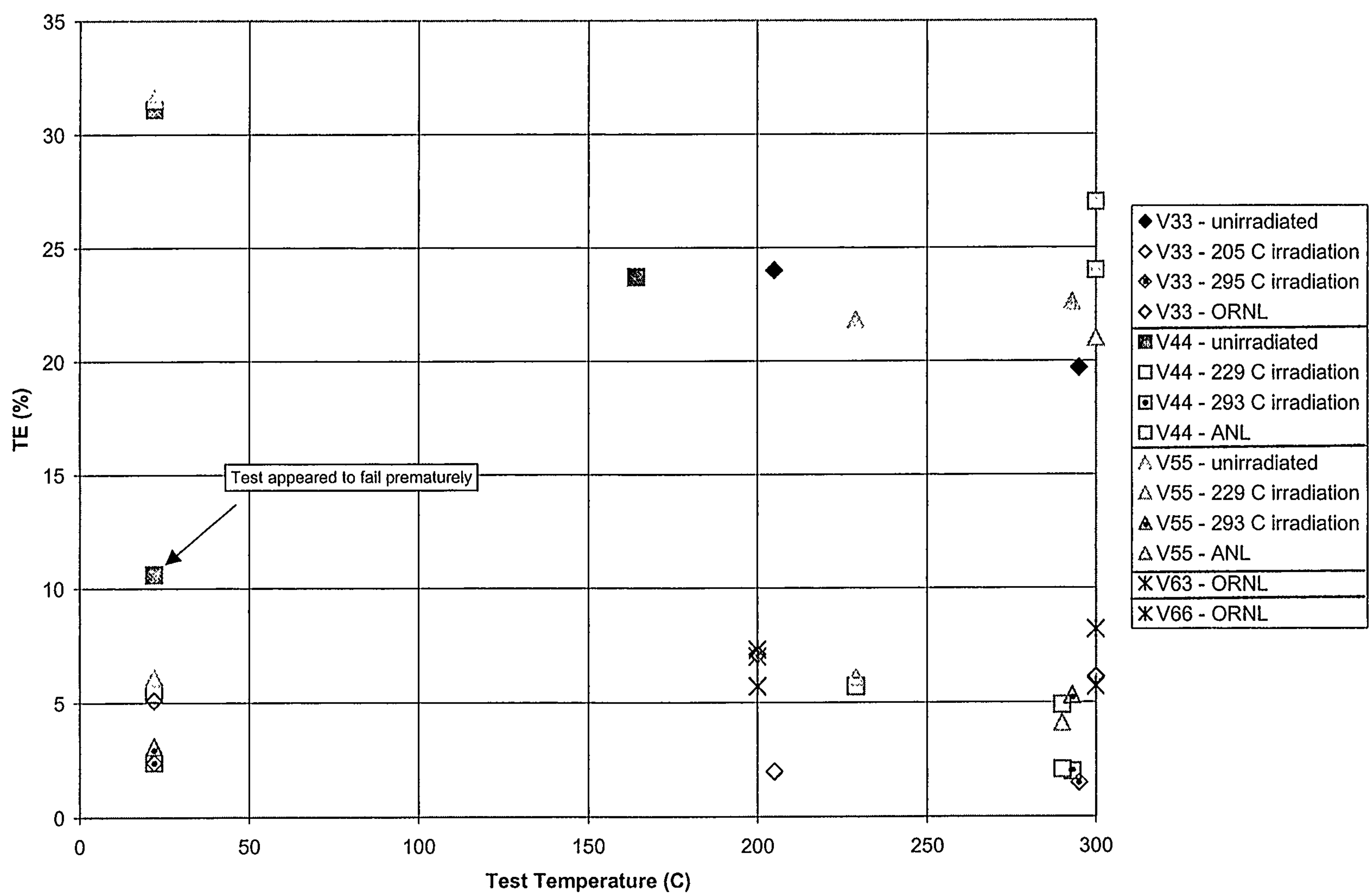

Figure 4. Total elongation of vanadium alloys irradiated in ATR.

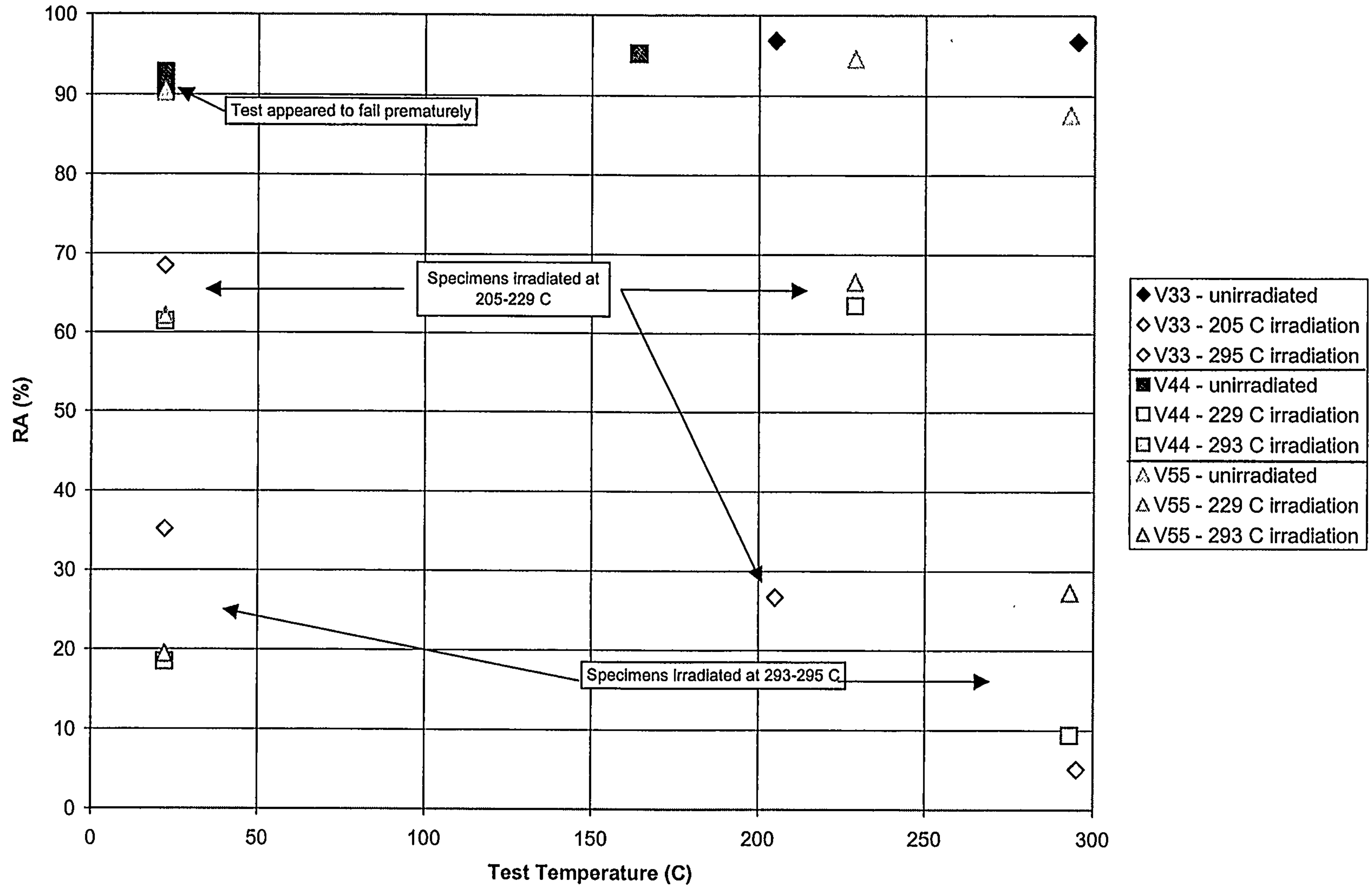

Figure 5. Reduction of area in vanadium alloys irradiated in ATR.

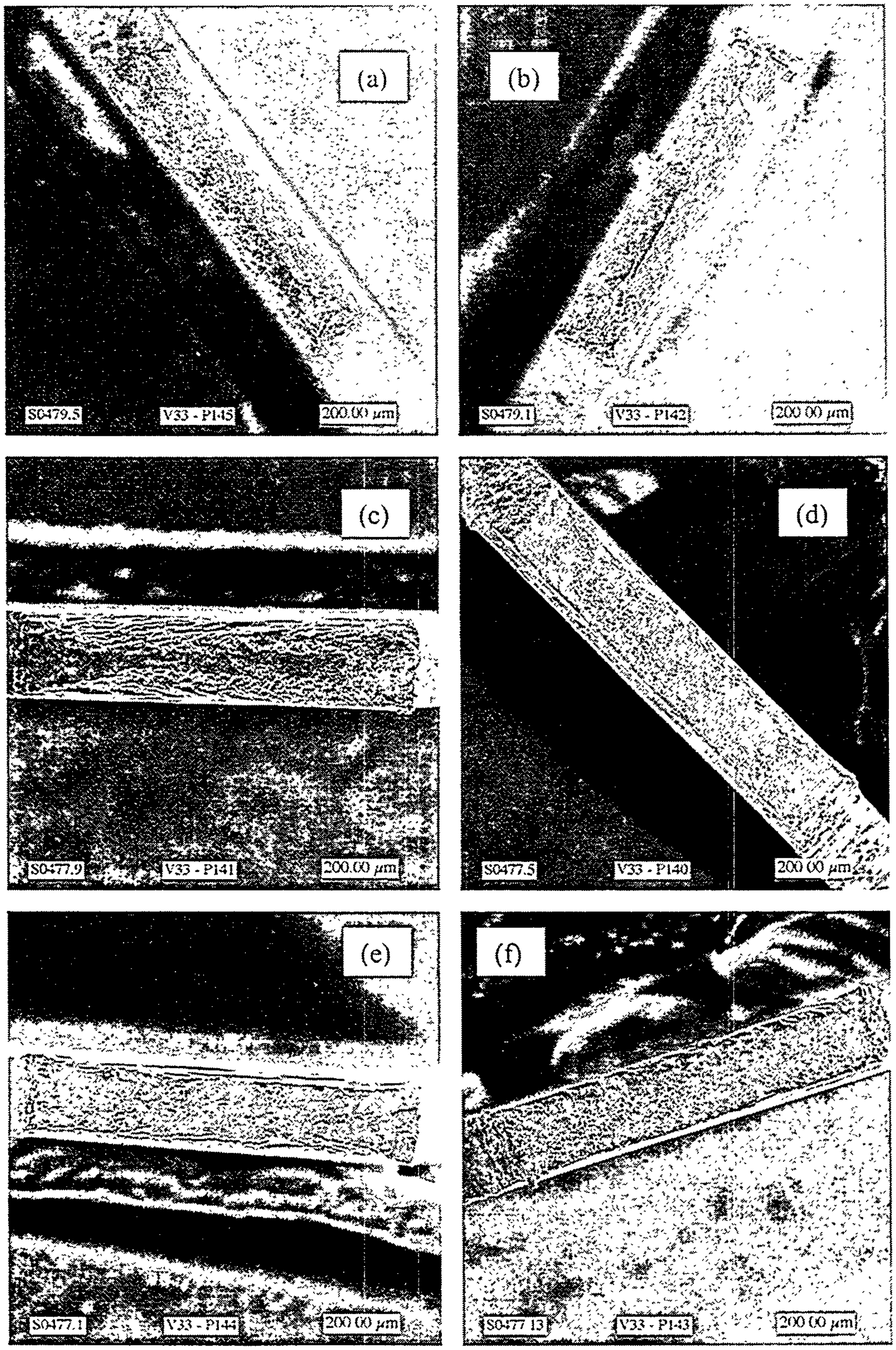

Figure 6a-f. Fractographs of alloy V33 specimens a)P145, b)P142, c)P141, d)P140, e)P144, and f)P143.

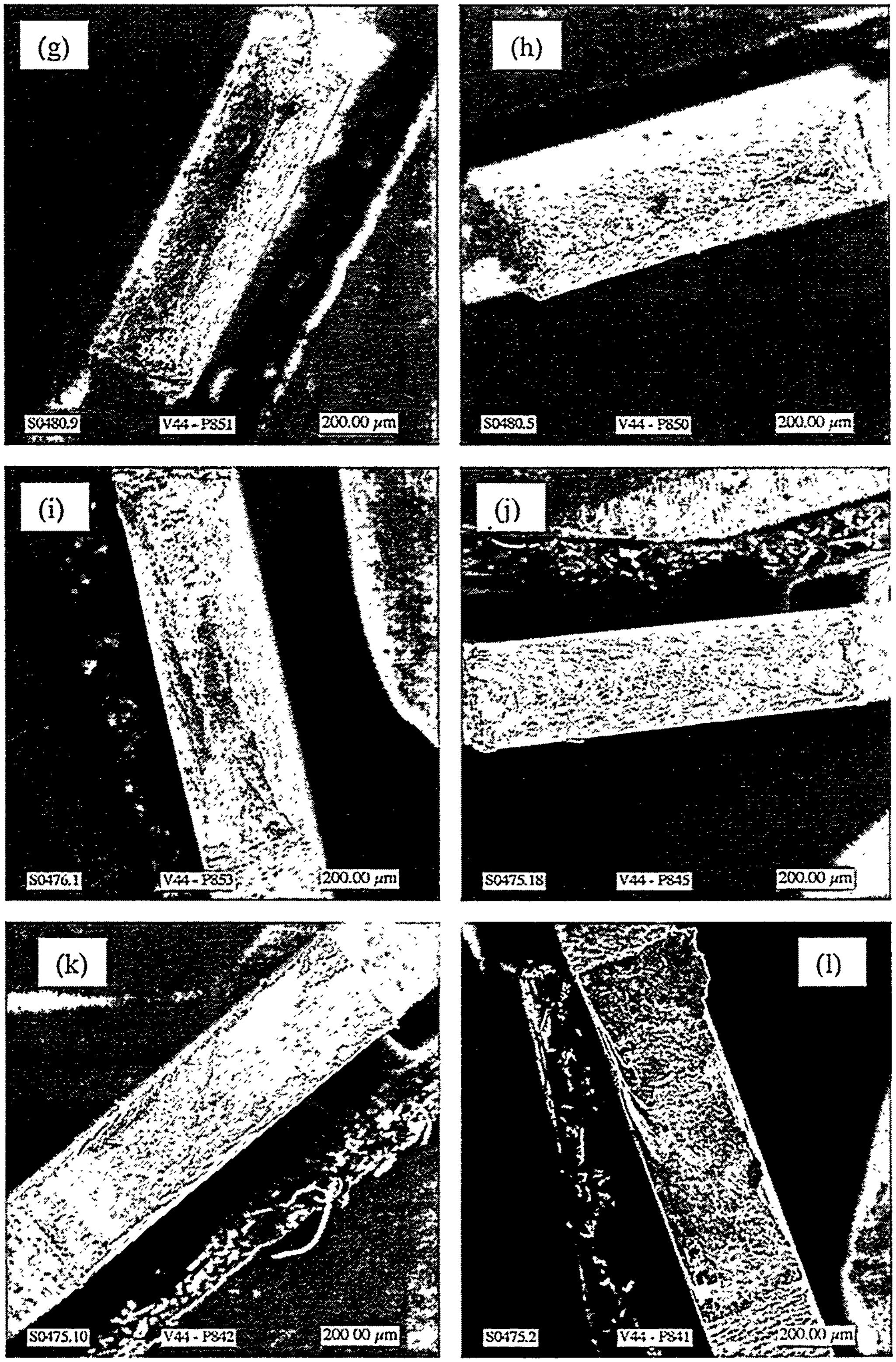

Figure 6g-l. Fractographs of V44 alloy specimens g)P851, h)P850, i)P853, j)P845, k)P842, and l)P841.

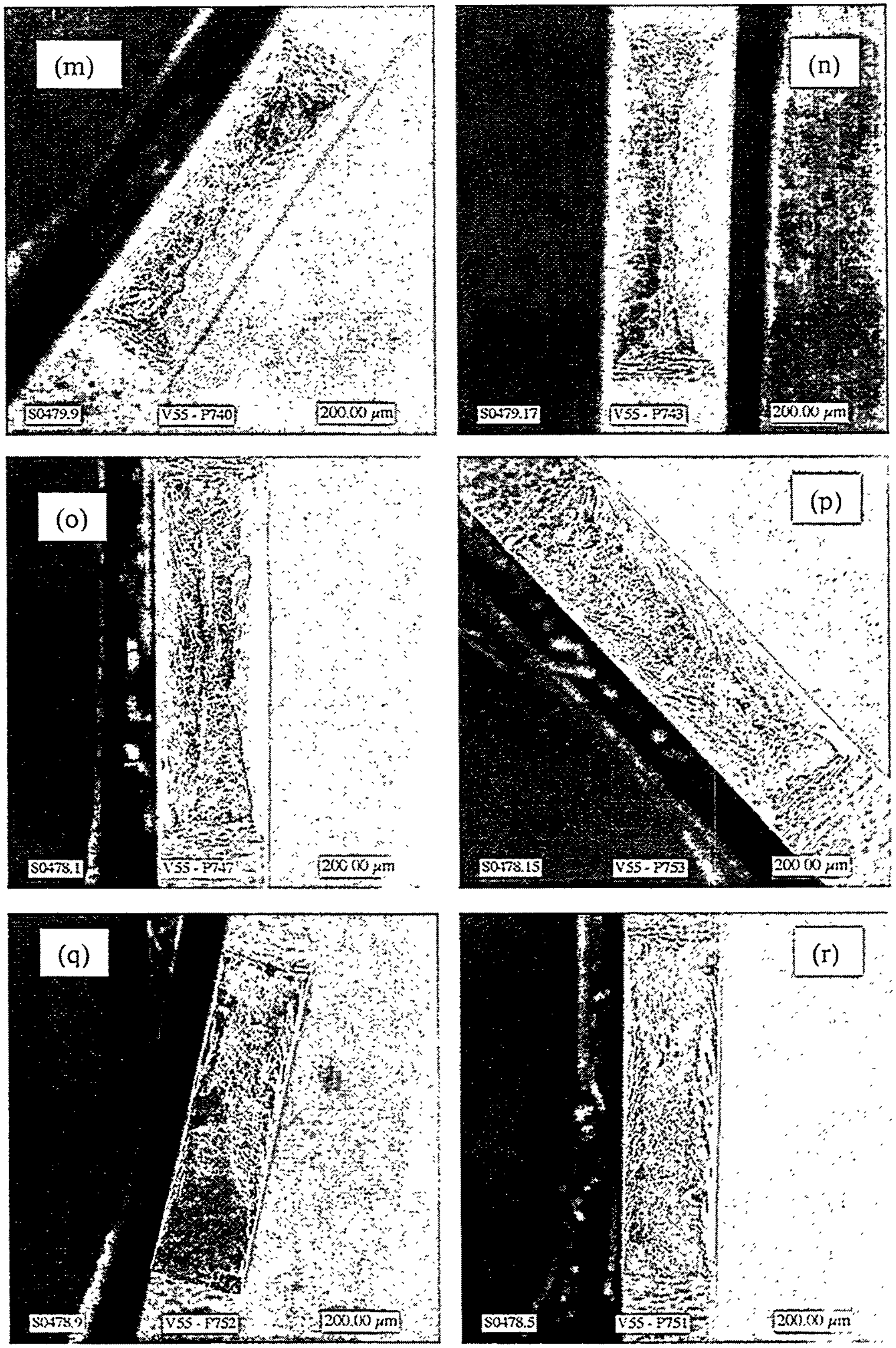

Figure 6m-r. Fractographs of V55 alloy specimens m)P740, n)P743, o)P747, p)P753, q)P752, and r)P751.

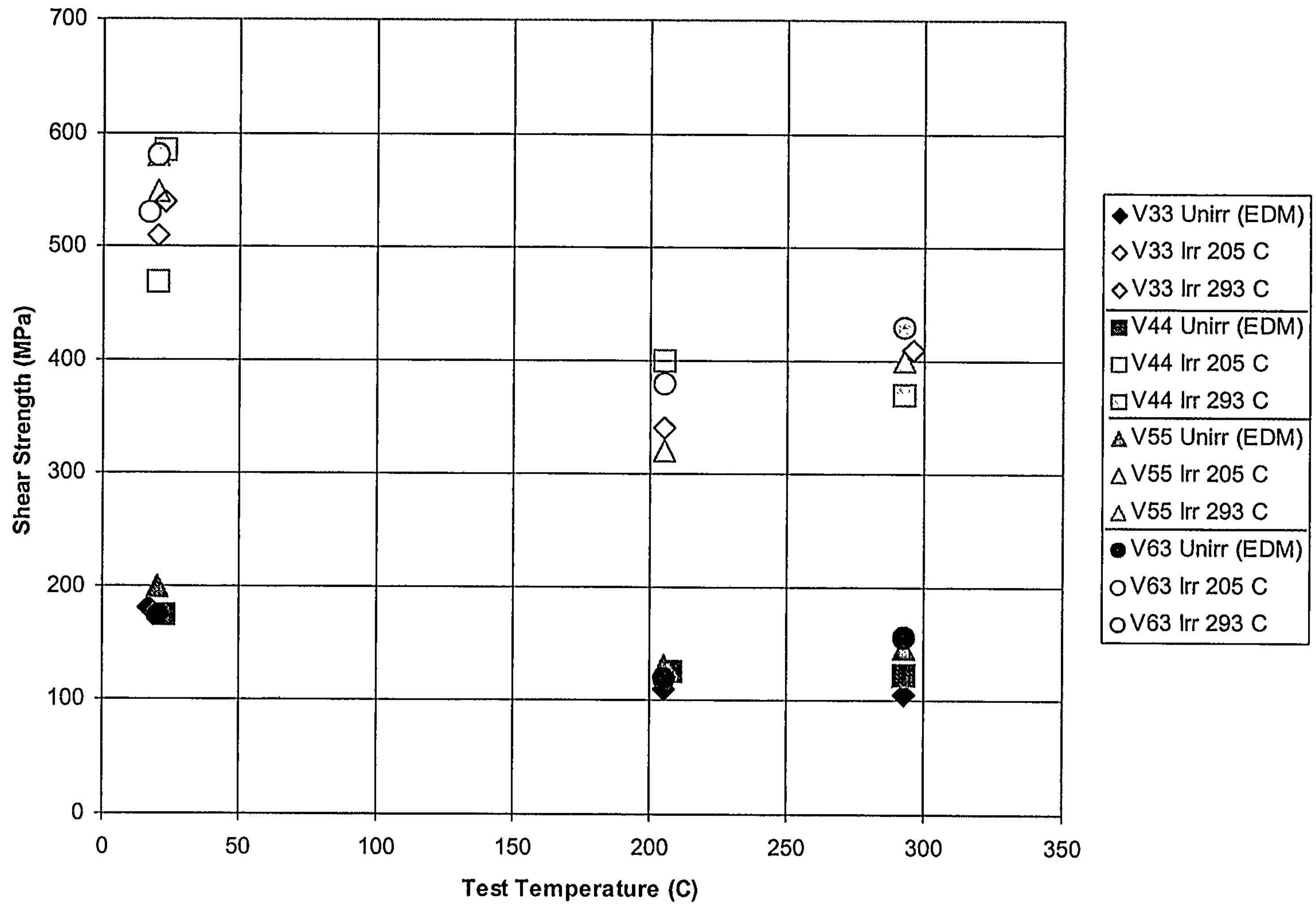

Figure 7. Effective shear yield strength in vanadium alloys irradiated in ATR.

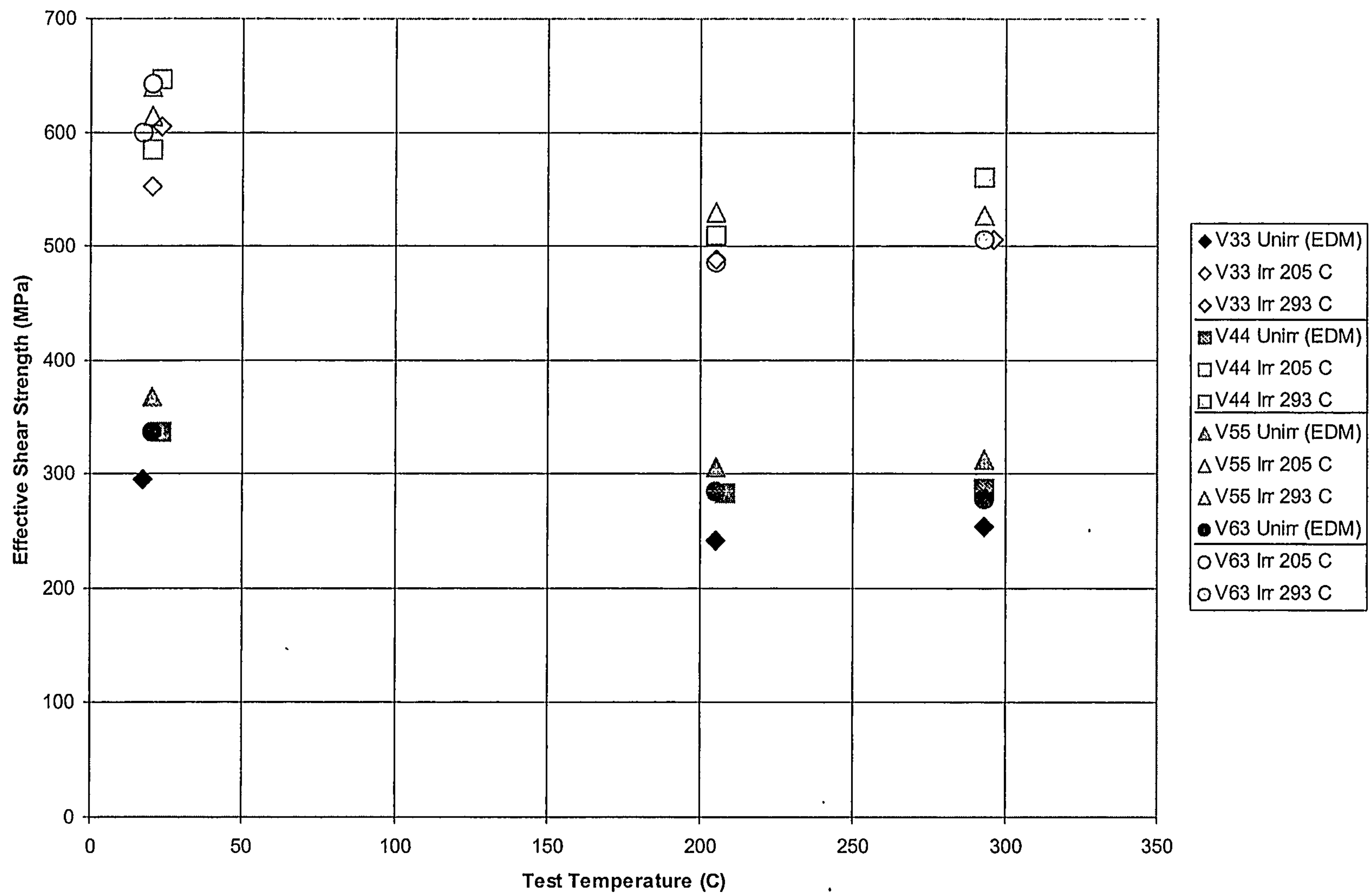

Figure 8. Effective maximum shear strength in vanadium alloys irradiated in ATR.

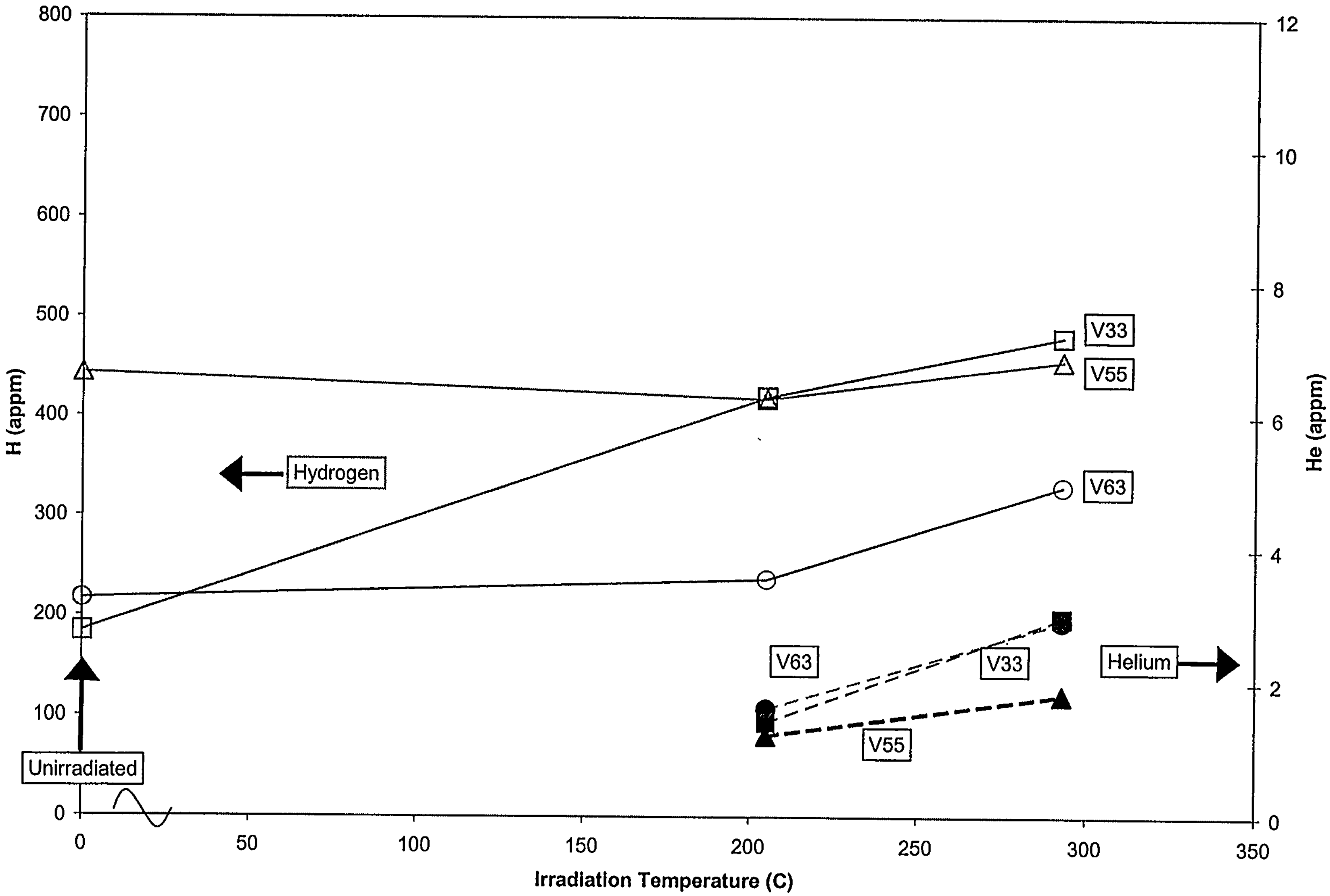

Figure 9. Gas content of selected vanadium alloy specimens irradiated in ATR.

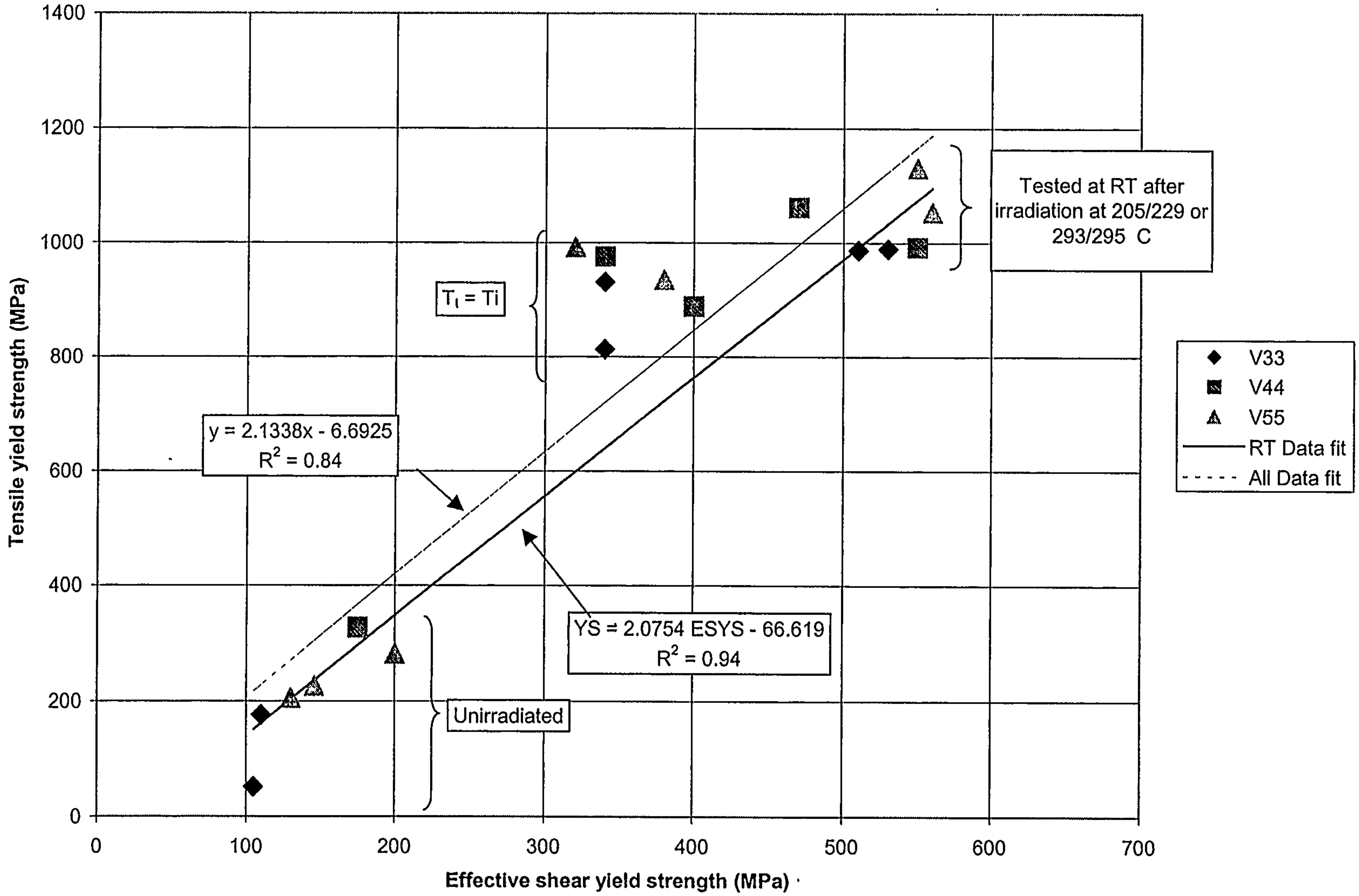

Figure 10. Correlation between tensile yield and effective shear yield strength for vanadium alloys irradiated in ATR.

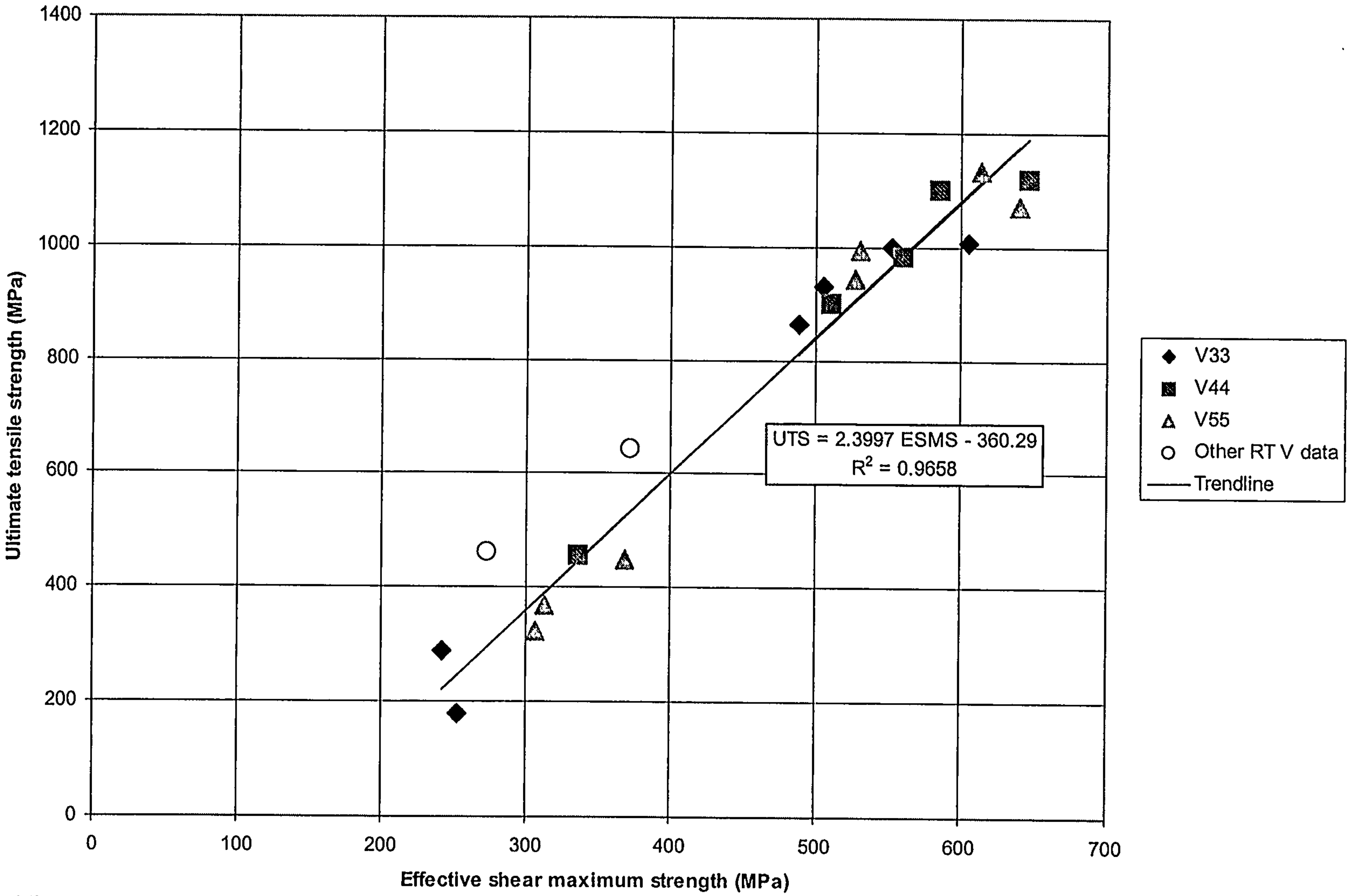

Figure 11. Correlation between maximum tensile and effective shear maximum strength for vanadium alloys irradiated in ATR.