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STRENGTH, DUCTILITY, AND DUCTILE-BRITTLE TRANSITION TEMPERATURE FOR MFR CANDIDATE VANADIUM ALLOYS*

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STRENGTH, DUCTILITY, AND DUCTILE-BRITTLE TRANSITION TEMPERATURE FOR MFR CANDIDATE VANADIUM ALLOYS

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Abstract

The dependence of the yield strength, tensile strength, elongation, and reduction in area on temperature for the V-15Ti-7.5Cr, V-20Ti, V-15Cr-5Ti, V-12Cr-5Ti, V-10Cr-5Ti, and V-3Ti-1Si alloys was determined from tensile tests at temperatures ranging from 25 to 700°C. The strength of the alloys increased with an increase of the combined Cr and Ti concentration. The total elongation for the alloys ranged between 20% and 38%. The reduction in area ranged from 30% to 90%. The DBTT, which was determined from the temperature dependence of the reduction in area, was less than 25°C for the V-15Ti-7.5Cr, V-20Ti, and V-3Ti-1Si alloys. The DBTT for the V-10Cr-5Ti, V-12Cr-5Ti, and V-15Cr-5Ti alloys was also less than 25°C if these alloys were annealed to reduce the hydrogen concentration prior to the tensile test. If these latter alloys were not annealed prior to the tensile test, the DBTT ranged from 40°C to 90°C and the DBTT increased with an increase of the Cr concentration. A Cr/Ti concentration ratio of 0-0.5 in these alloys was found to cause the alloys to be less susceptible to hydrogen embrittlement.

1. Introduction

The advantages of the vanadium-base alloys over other candidate alloys for structural materials purposes in a magnetic fusion reactor (MFR) have been previously cited in the literature [1-3]. Vanadium is inherently a low-activation material on exposure to a neutron flux and its low neutron absorption characteristics minimize its impact on tritium breeding. In addition, vanadium alloys possess high thermal conductivities and low thermal expansion coefficients which would result in low thermal stresses for a given heat flux. Neutron irradiations to 40 atom displacements per atom (dpa) and ion irradiations to 200 dpa of vanadium alloys containing at least 3 wt % Ti have demonstrated an inherent resistance of these alloys to void and cavity swelling [4,5]. The available information suggests that the vanadium alloys are generally compatible with liquid lithium which may be used as a coolant in a MFR. However, recent experimental results have shown that some vanadium alloys, e.g., the V-15Cr-5Ti alloy, become embrittled during moderate fluence (40 dpa) neutron irradiation while other alloys do not become embrittled, e.g., the V-20Ti and V-3Ti-1Si alloys [5]. These latter results suggest that the optimization of the vanadium alloy composition for acceptable mechanical properties in a MFR environment may not yet be complete. To optimize the use of available reactor space for neutron irradiation of mechanical property specimens of candidate vanadium alloys, it was considered expedient to evaluate some of the alloys on the basis of the tensile properties for the materials in the unirradiated condition. This paper presents the tensile properties of several candidate vanadium alloys. The results indicate that certain V-Cr-Ti alloys may be more susceptible to hydrogen embrittlement as structural materials for a MFR.

2. Materials and procedure

Vanadium alloys with nominal compositions of V-15Ti-7.5Cr, V-20Ti, V-15Cr-5Ti, V-12Cr-5Ti, V-10Cr-5Ti, and V-3Ti-1Si were obtained in the form of

~50% cold-worked sheet with an approximate thickness of 0.9 mm. Chemical analyses of the materials, that were performed by the Analytical Department of the Teledyne Wah Chang Albany Company, are presented in Table 1. Tensile specimens with the dimensions shown in Fig. 1 were machined from the as-received sheet. Specimens were machined with the tensile axis orientation either parallel to the rolling direction or perpendicular to the rolling direction of the as-received sheet. Since the tensile properties were not significantly different for the two orientations, an orientation distinction will not be considered in this paper. The tensile specimens of the 50% cold-worked alloys were recrystallized by annealing for 1 h in a vacuum of 2×10^{-5} Pa. The V-15Ti-7.5Cr, V-15Cr-5Ti, V-12Cr-5Ti, and V-10Cr-5Ti alloys were annealed at 1125°C. The annealing temperatures for the V-20Ti and V-3Ti-1Si alloys were 1100°C and 1050°C, respectively. These annealing temperatures resulted in an average, recrystallized grain diameter of ~0.020 mm. The surfaces of the tensile specimens were mechanically ground and polished to a surface finish of ~0.3 μm. During the surface preparation, the specimens were exposed to water, ethylene glycol, acetone, ethyl alcohol, and methyl alcohol. The as-machined tensile specimens were either annealed and then mechanically polished (AP) or mechanically polished and subsequently annealed (PA) prior to tensile testing. In this study, the majority of the specimens were strained to fracture after using the (AP) procedure. The (AP) procedure was believed to more closely simulate the exposure of vanadium alloys to various environments, e.g., liquid ammonia, ethyl alcohol, and water, after corrosion testing or irradiation in liquid alkali metals. The release of hydrogen from the tensile specimens during annealing was determined by use of a quadrupole, partial-pressure gas analyzer mounted in the ion-pumped vacuum system.

The tensile specimens were tested at a tensile strain rate of 0.0011 s^{-1} for a crosshead speed of 0.008 $mm \cdot s^{-1}$. All of the tensile tests were conducted in an environment of flowing (3×10^{-5} $m^3 \cdot s^{-1}$) argon of 99.9999% purity. The temperature of the specimens was determined by use of a chromel-alumel thermocouple that was arc-welded to the edge of the specimen, and the temperature of the specimen during the tensile test was controlled to ±1°C. The tensile specimens were heated to the test temperature at a rate of 0.2°C$\cdot s^{-1}$.

3. Experimental results

The yield strength (Y.S.) evaluated at 0.2% offset or the yield point, the ultimate tensile strength (U.T.S.), uniform elongation (E_u), total elongation (E_t), and reduction in cross-sectional area (R.A.) for the alloys at 25°C to 700°C are listed in Table 2. The temperature dependence of the Y.S., U.T.S., and R.A. is shown in Figs. 2, 3, and 4, respectively. For clarity in Figs. 2, 3, and 4, data are only shown for the V-15Ti-7.5Cr (ANL 94), V-15Cr-5Ti (ANL 101), and V-3Ti-1Si (ORNL 10837) alloys. In the case of the V-20Ti alloy, a curve in Figs. 2 and 3 for the temperature dependence of the strength would be displaced ~50 MPa below the curve for the V-15Ti-7.5Cr alloy. In the case of the V-10,12Cr-5Ti alloys and the V-15Cr-5Ti (CAM 834 and ANL 204) alloys, curves for their temperature dependence of the strength would be ±50 MPa from the curve for the V-15Cr-5Ti (ANL 101) alloy. The dependence of the ratio of the hydrogen partial pressure (P_H) to the total gas pressure (P_T) in the vacuum system on temperature during heating (0.2°C$\cdot s^{-1}$) of the as-polished V-12Cr-5Ti tensile specimens is also shown in Figs. 2, 3, and 4. The peak in the P_H/P_T spectrum was lower by approximately one order of magnitude for alloy specimens that were annealed in the as-received condition.

The yield strength and tensile strength for the alloys increased with an increase of the combined Cr and Ti concentration (Figs. 2 and 3), and the strength of the alloys decreased with an increase of the test temperature from 25°C to 225°C for both the (AP) and (PA) procedures. The yield and tensile strength for an alloy with the (PA) procedure was significantly lower than the yield and tensile strength for an alloy with the (AP) procedure for test temperatures of 25°C and 100°C. The yield strength of the alloys was essentially constant at test temperatures between 225°C and 600°C; however, the tensile strength for the alloys increased on increasing the test temperature from ~225°C to 420°C. The experimental results suggest that the yield and tensile strength for an alloy were not dependent on the (AP) or (PA) procedure at temperatures above 225°C. The yield and tensile strength for the V-15Ti-7.5Cr, V-20Ti, V-10,12Cr-5Ti, and V-3Ti-1Si alloys decreased significantly above 600°C whereas the yield and tensile strength for the V-15Cr-5Ti alloys did not decrease above 600°C. The onset of the temperature-independent yield stress regime at 225°C was also the temperature for the onset of a significant release of hydrogen during annealing of an alloy with the (AP) or (PA) procedure. It should be noted that, with the exception of a V-12Cr-5Ti and V-15Cr-5Ti (ANL 101) specimens at 225°C, tensile specimens with the (AP) procedure were tested at temperatures ranging from 225°C to 700°C. Moreover, the heating rate to the tensile test temperature was approximately the same as the heating rate for the generation of the data on the release of hydrogen. The increase of the tensile strength for the alloys at test temperatures above 225°C can also be correlated with a pronounced increase in the release of hydrogen. A yield point of varying magnitude was evident in the load-elongation curve for each of the alloys on testing at temperatures below 425°C. For test temperatures of 425°C and above, the yield point was replaced by a serrated yielding. The onset of serrated yielding can be correlated with the abrupt decline of P_H/P_T at 425°C (Fig. 2). Serrations in the load-elongation curves for deformation beyond the initial yielding were evident for the alloys on testing at temperatures ranging from 325°C to 600°C (Fig. 3).

The total elongation for the alloys ranged from 20% to 38%, and the values were not strongly dependent on the test temperature (Table 2). In contrast, the reduction in cross-sectional area (R.A.), which ranged from 32% to 90%, was strongly dependent on the test temperature (Fig. 3) and also on the (AP) and (PA) procedure. In Fig. 3, an R.A. curve for the V-20Ti alloy would closely parallel the curve for the V-15Ti-7.5Cr alloy. Also, R.A. curves for the V-10,12Cr-5Ti alloys and the V-15Cr-5Ti (CAM 834 and ANL 204) alloys would parallel the curve for the V-15Cr-5Ti (ANL 101) alloy. The R.A. for the alloys with the (AP) procedure increased with an increase of test temperature from 25°C to 225°C. For test temperatures above 225°C, the R.A. for alloys with the (AP) procedure either decreased as the test temperature was increased to 600°C, viz., the V-20Ti and V-15Ti-7.5Cr alloys, or remained essentially constant to ~520°C and then decreased with an increase of test temperature, viz., V-10,12,15Cr-5Ti and V-3Ti-1Si alloys. The experimental results suggest that the R.A. for an alloy was not dependent on the (AP) or (PA) procedure for test temperatures above 225°C. The R.A. for the alloys at 25°C increased substantially with a change of the procedure from (AP) to (PA). The increase of the R.A. at 25°C was especially pronounced (30-40% to 80-90%) for the V-10,12,15Cr-5Ti alloys. In contrast, the increase of the R.A. at 25°C for the V-15Ti-7.5Cr, V-20Ti, and V-3Ti-1Si alloys was substantially less, e.g., 64% to 75% for the V-15Ti-7.5Cr alloy.

Optical microscopy of the fracture surfaces of the tensile specimens suggested that the eventual fracture of the tensile specimens was determined by the dominence of either transgranular cleavage or ductile elongation (tearing). The R.A. was considered to be the most accurate parameter determined from the tensile tests for a quantitative evaluation of the dominence of transgranular cleavage and ductile elongation. We have arbitrarily taken the 55% R.A. as the transition from a dominence of the transgranular cleavage to a dominence of ductile elongation. On this basis, the ductile-brittle transition temperatures (DBTT) for the alloys that underwent the (AP) or (PA) procedure are listed in Table 3. All of the alloys with the (PA) procedure had a DBTT below 25°C. In the case of the alloys that underwent the (AP) procedure, the DBTT for the V-15Ti-7.5Cr, V-20Ti, and V-3Ti-1Si alloys was less than 25°C, whereas the DBTT for the V-10,12,15Cr-5Ti alloys ranged from 40 to 90°C. The DBTT for the V-10,12,15Cr-5Ti alloys with the (AP) procedure increased with an increase of the Cr concentration in the alloy.

4. Discussion of results

The addition of either chromium, titanium, hydrogen, oxygen, nitrogen, or carbon to pure vanadium, in general, produces solid solution strengthening, i.e., an increase of the yield and tensile strength and a decrease of ductility [6-8]. The results of the present study show that the strength of the V-15Ti-7.5Cr, V-20Ti, V-15Cr-5Ti, V-12Cr-5Ti, V-10Cr-5Ti, and V-3Ti-1Si alloys at temperatures from 25°C to 700°C increased with an increase of the combined Cr and Ti concentration.

The presence of hydrogen impurity in the alloys had an especially significant effect on the tensile properties of the alloys. The increase in the yield and tensile strength, the decrease in the reduction of the cross-sectional area, and the increase of the DBTT for the alloys on changing the procedure for the preparation of the tensile specimens from (PA) to (AP) can be attributed to an increase of the hydrogen concentration in the tensile specimens during the (AP) procedure. It was not established if the hydrogen was introduced into the tensile specimens by mechanical abrasion during grinding and polishing and/or by contact with water and organic media. The embrittlement of vanadium upon contact with water and organic media has been attributed to an increase in hydrogen concentration [9,10].

Crack initiation and propagation in a vanadium alloy containing hydrogen impurity can be related to the hydrogen solubility and diffusivity in the alloy [11]. A decrease in the hydrogen solubility and/or an increase in the hydrogen diffusivity in vanadium by the addition of alloying elements can be expected to increase the tendency for crack propagation and embrittlement [10]. Titanium increases the solubility of hydrogen in vanadium and chromium decreases the solubility [11,12]. The diffusivity of hydrogen in vanadium decreases with the addition of titanium [11]. Therefore, the greater susceptibility of the V-10,12,15Cr-5Ti alloys to hydrogen embrittlement in comparison with the V-15Ti-7.5Cr, V-20Ti, V-3Ti-1Si alloys with the (AP) procedure can be correlated with the higher Cr/Ti concentration ratio for the former group in comparison with the latter group, i.e., a Cr/Ti ratio of 2-3 for the former group and 0-0.5 for the latter group.

The presence of oxygen (nitrogen and carbon) impurity in the alloys also had a significant effect on the strength of the alloys. The onset of serrations in the load-elongation curves was clearly correlated with either the decrease of the yield strength to a plateau value (Fig. 2) or the significant increase of the tensile strength (Fig. 3). Serrated yielding and deformation is generally attributed to the diffusion of impurity atoms, e.g., oxygen, nitrogen, and carbon, to moving dislocations which, in turn, repeatedly break away from their impurity atmospheres. Alternatively, the dislocations formed during the deformation collect solute atmospheres and remain locked in position so that additional dislocation sources are activated with the resultant formation of repeated minute yield points [13]. The onset of serrations in the load-elongation curves obtained in this study may also be correlated with the removal of hydrogen from the tensile specimens. This correlation suggests that oxygen (nitrogen and carbon) impurity atoms may be strong traps for hydrogen atoms [14]. Therefore, the concentration of oxygen (nitrogen and carbon) impurity in an alloy may have been an additional factor that determined the susceptibilty of an alloy to hydrogen embrittlement.

5. Conclusions

The yield and tensile strength for the V-15Ti-7.5Cr, V-20Ti, V-15Cr-5Ti, V-12Cr-5Ti, V-10Cr-5Ti, and V-3Ti-1Si alloys increases with an increase of the combined Cr and Ti concentration. The total elongation, that is determined from tensile tests on these alloys at 25°C to 700°C, ranges from 20% to 38%. The V-Cr-Ti alloys with a Cr/Ti concentration ratio of 2-3, i.e., the V-10,12,15Cr-5Ti alloys, are more susceptible to hydrogen embrittlement than V-Cr-Ti alloys with a Cr/Ti concentration ratio of 0-0.5, i.e., the V-15Ti-7.5Cr, V-20Ti, and V-3Ti-1Si alloys. The DBTT, which is determined from the temperature dependence of the reduction in area, is less than 25°C for the V-15Ti-7.5Cr, V-20Ti, and V-3Ti-1Si alloys. The DBTT for the V-10Cr-5Ti, V-12Cr-5Ti, and V-15Cr-5Ti alloy is also less than 25°C if these alloys are annealed to reduce the hydrogen concentration prior to the tensile test. If these latter alloys are not annealed prior to the tensile test, the DBTT ranges from 40°C to 90°C and the DBTT increases with an increase of the Cr concentration. The exposure of vanadium-base alloys to hydrogenous media during specimen preparation and/or after exposure to other environments (e.g., liquid alkali metals) can have a significant effect on the mechanical properties because of hydrogen pickup by the materials. The mechanical testing environment (e.g., vacuum or inert gas) as well as the test conditions (i.e., temperature, heating rate), which influence hydrogen egress from the material, are also important parameters in the subsequent mechanical behavior.

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Table 1. Alloy Compositions

Nominal Composition	Melt Number	Concentration (wt %)		Concentration (ppm)									
		Cr	Ti	O	N	C	Si	H	S	Nb	Mo	Fe	Al
V-15Ti-7.5Cr	ANL 94	7.2	14.5	1110	250	400	400	<5	20	110	160	910	30
V-20Ti	CAM 832	-	17.7	830	160	380	480	<5	<10	44	200	390	33
V-15Cr-5Ti	CAM 834	12.9	5.9	400	490	280	1230	9	50	50	320	420	150
V-15Cr-5Ti	ANL 204	14.5	5.0	330	96	120	400	<5	20	33	87	180	210
V-15Cr-5Ti	ANL 101	13.5	5.2	1190	360	500	390	<5	<30	100	540	520	40
V-12Cr-5Ti	TWC 040	10.9	5.0	470	80	90	270	<5	20	28	140	350	140
V-10Cr-5Ti	ANL 206	9.2	4.9	230	31	100	340	<5	20	160	<20	170	140
V-3Ti-1Si	ORNL 10837	-	3.1	210	310	310	2500	8	120	55	270	380	160

Table 2. Temperature Dependence of Tensile Properties for Vanadium Alloys

Alloy	T (°C)	Y.S. (MPa)	U.T.S. (MPa)	E_u (%)	E_t (%)	R.A. (%)
V-15Ti-7.5Cr (ANL 94)	25[a]	636	717	19.9	28.3	75.4
	25	643	718	21.0	29.7	63.7
	100[a]	556	647	20.5	28.2	77.1
	100	602	688	20.7	27.3	66.4
	225	490	618	19.7	24.7	73.5
	325	493	682	20.7	25.2	69.3
	420	485	730	22.0	29.0	66.5
	520	484	730	24.1	27.6	61.5
	600	469	738	25.7	27.1	51.0
	650	441	661	23.1	27.6	52.8
	700	350	584	18.1	30.3	52.8
V-20Ti (CAM 832)	25[a]	599	655	27.8	30.7	74.7
	25	628	692	21.9	32.1	67.5
	100[a]	525	640	22.0	30.1	76.0
	225	437	588	19.9	24.5	65.8
	325	436	636	23.9	28.5	68.9
	420	443	658	24.4	29.7	59.5
	520	445	678	28.6	33.1	58.5
	600	417	667	20.2	28.0	55.1
	650	400	554	20.2	29.4	71.7
	700	331	496	13.4	22.7	66.8
V-15Cr-5Ti (CAM 834)	25[a]	533	627	24.9	31.0	85.4
	25	588	688	25.5	27.8	32.2
	100	483	589	24.9	36.6	58.5
	225	407	548	22.6	29.1	81.7
	325	368	522	23.2	29.7	85.4
	420	379	571	21.4	26.6	77.7
	520	359	567	20.2	26.8	72.3
	600	384	610	25.3	31.1	71.3
	650	373	573	17.6	30.2	70.3
	700	355	519	20.6	32.4	72.8
V-15Cr-5Ti (ANL 204)	25[a]	520	624	24.5	31.5	82.5
	25	570	674	25.8	32.3	40.3
	100	495	599	22.8	28.6	61.2
	225	399	525	22.0	27.3	82.2
	325	348	500	18.8	25.6	81.4
	420	346	524	20.4	26.0	79.8
	520	350	538	19.2	24.5	69.9
	600	335	560	20.9	24.8	66.2
	650	356	575	22.3	27.4	63.0
	700	339	564	21.5	28.1	61.6

Table 2. (Contd.)

Alloy	T (°C)	Y.S. (MPa)	U.T.S. (MPa)	E_u (%)	E_t (%)	R.A. (%)
V-15Cr-5Ti (ANL 101)	25[a]	532	632	23.3	32.1	80.2
	25	579	682	24.7	32.9	43.3
	100[a]	439	545	22.0	31.1	85.9
	100	485	593	24.1	32.0	65.4
	225[a]	365	470	22.1	28.1	85.0
	225	370	478	17.1	24.9	86.4
	325	317	449	16.7	23.2	80.9
	420	341	518	20.7	26.6	83.2
	520	326	502	16.9	22.6	74.6
	600	342	555	20.7	26.0	71.6
	650	342	559	24.8	30.3	61.2
	700	337	544	20.9	25.7	60.4
V-12Cr-5Ti (TWC 040)	25[a]	491	573	23.1	31.0	84.6
	25	519	613	26.4	31.1	40.5
	100[a]	402	500	21.2	27.0	91.4
	100	442	539	21.0	28.9	66.5
	225	358	476	16.5	20.7	84.0
	225[a]	347	464	20.7	25.7	79.9
	325	304	448	17.3	23.9	82.0
	420	321	501	18.4	24.3	77.6
	520	303	486	17.2	22.0	71.5
	600	299	519	22.3	26.0	71.7
	650	310	529	20.2	24.5	65.6
	700	248	425	21.2	25.5	63.7
V-10Cr-5Ti (ANL 206)	25[a]	440	541	22.6	33.3	91.3
	25	521	617	26.5	32.3	47.5
	100[a]	387	482	20.3	31.5	90.8
	100	431	533	23.1	33.5	74.1
	225	334	448	21.5	28.7	82.4
	325	300	452	21.4	30.1	85.8
	420	300	467	19.7	25.2	83.2
	520	297	463	18.4	23.3	83.5
	600	297	502	21.3	24.5	78.1
	700	251	488	21.3	26.6	62.1
V-3Ti-1Si (ORNL 10837)	25[a]	360	468	29.1	22.3	87.2
	25	446	518	24.9	34.7	72.1
	100	352	464	21.9	27.4	80.2
	225	235	344	22.3	27.6	89.6
	325	227	386	17.2	24.0	90.4
	420	238	437	19.8	25.6	84.3
	520	228	424	19.2	23.0	87.4
	600	230	435	20.9	24.9	77.4
	700	160	330	21.5	26.5	67.5

[a]Alloy specimen underwent procedure (PA). Specimens without the superscript underwent the (AP) procedure.

Table 3. Ductile-Brittle Transition Temperatures (DBTT)

Alloy	Melt Number	DBTT (°C) (PA)	DBTT (°C) (AP)
V-15Ti-7.5Cr	ANL 94	<25	<25
V-20Ti	CAM 832	<25	<25
V-15Cr-5Ti	CAM 834	<25	90
V-15Cr-5Ti	ANL 204	<25	80
V-15Cr-5Ti	ANL 101	<25	60
V-12Cr-5Ti	TWC 040	<25	70
V-10Cr-5Ti	ANL 206	<25	40
V-3Ti-1Si	ORNL 10837	<25	<25

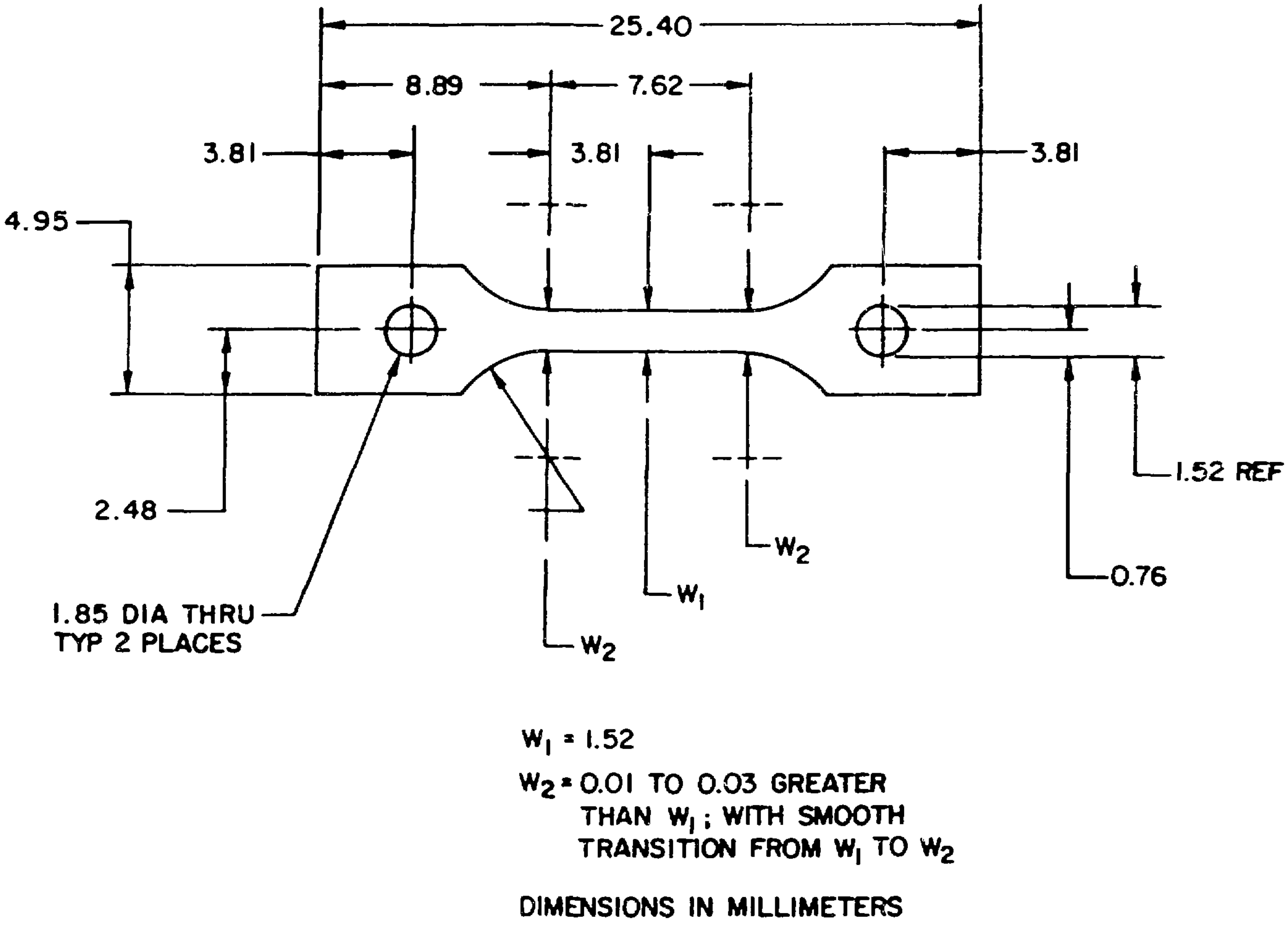

Fig. 1. Dimensions for Vanadium Alloy Tensile Specimens.

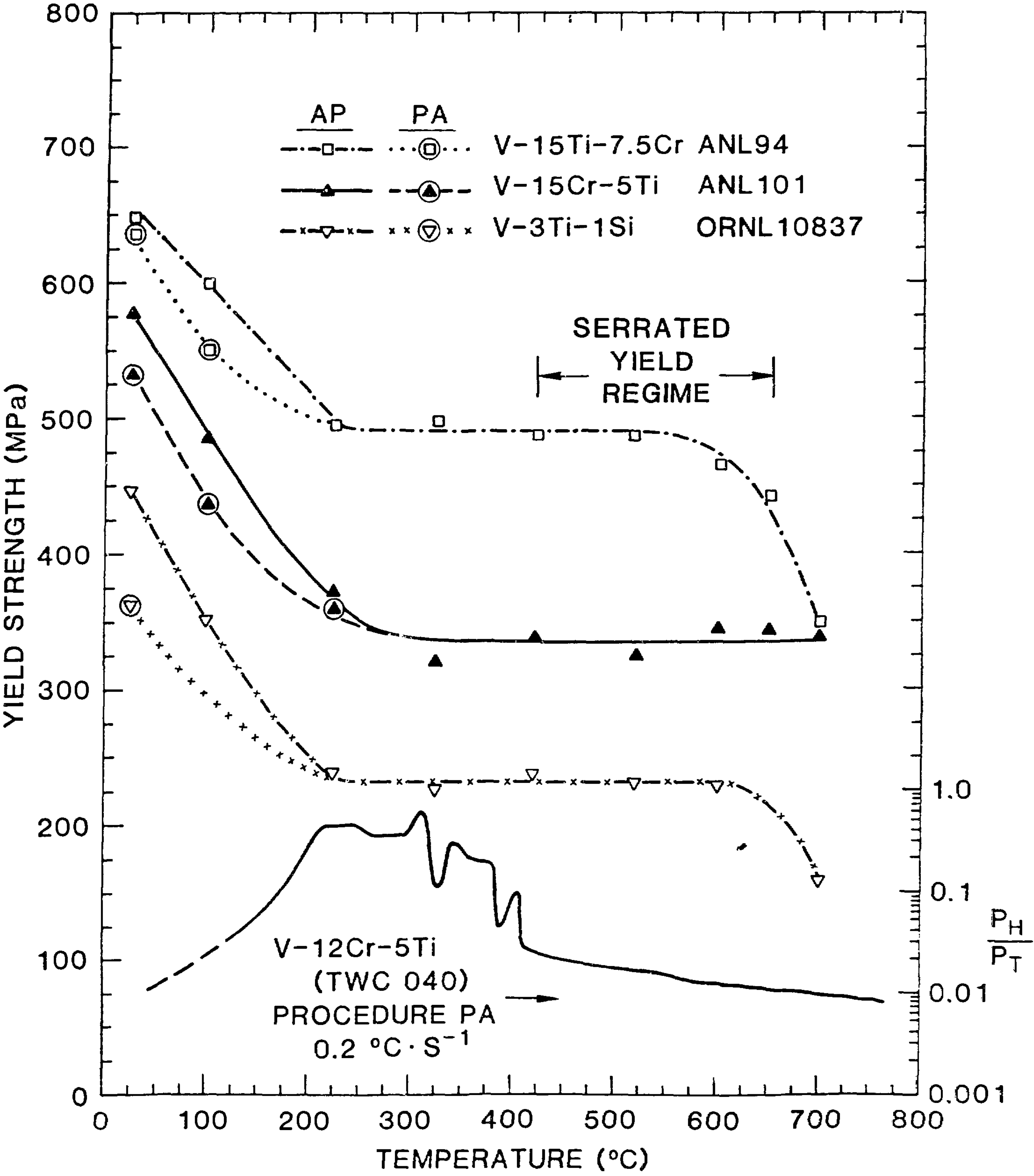

Fig. 2. Temperature Dependence of the Yield Strength and Hydrogen Evolution for Vanadium Alloys.

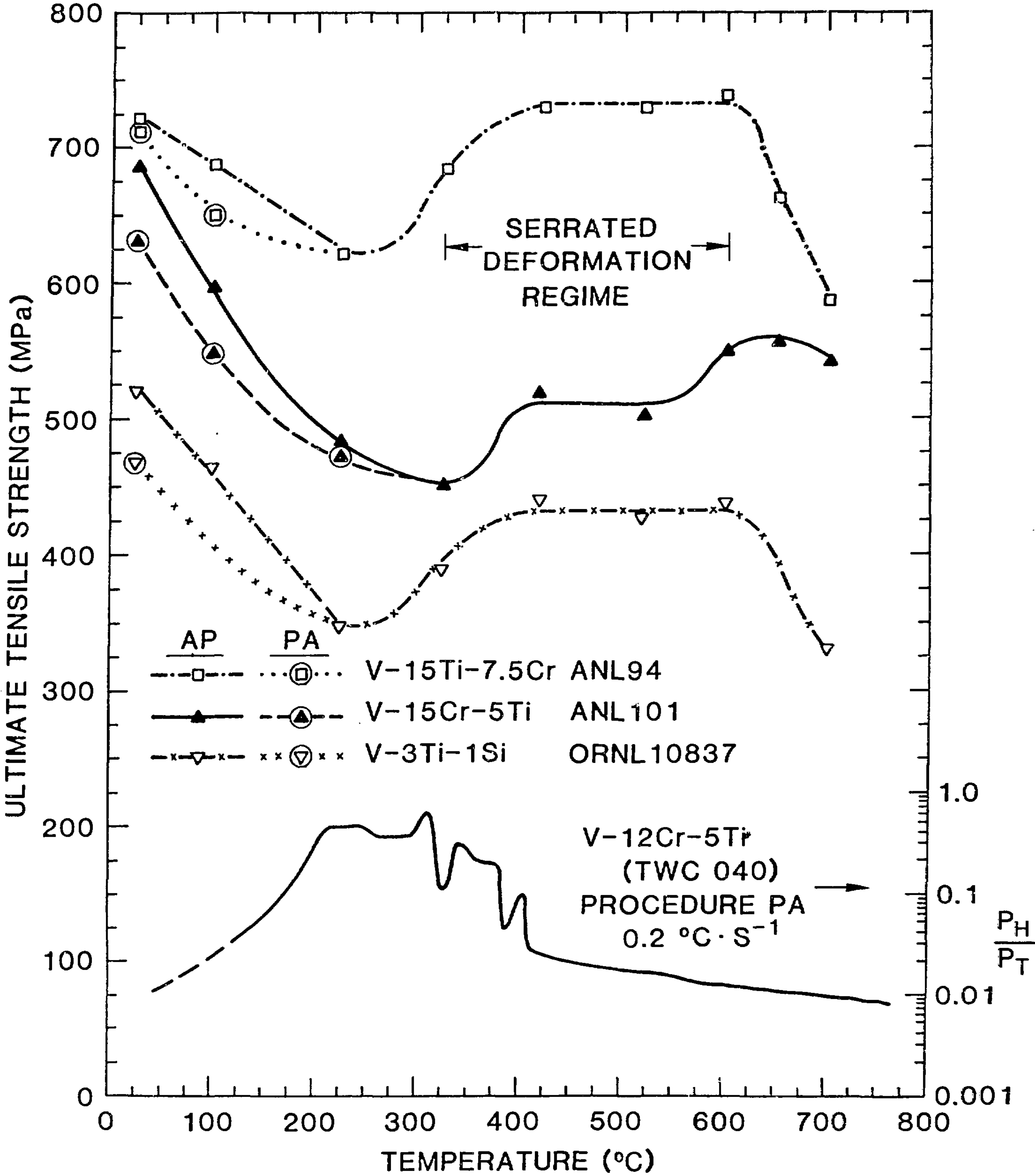

Fig. 3. Temperature Dependence of the Tensile Strength for Vanadium Alloys.

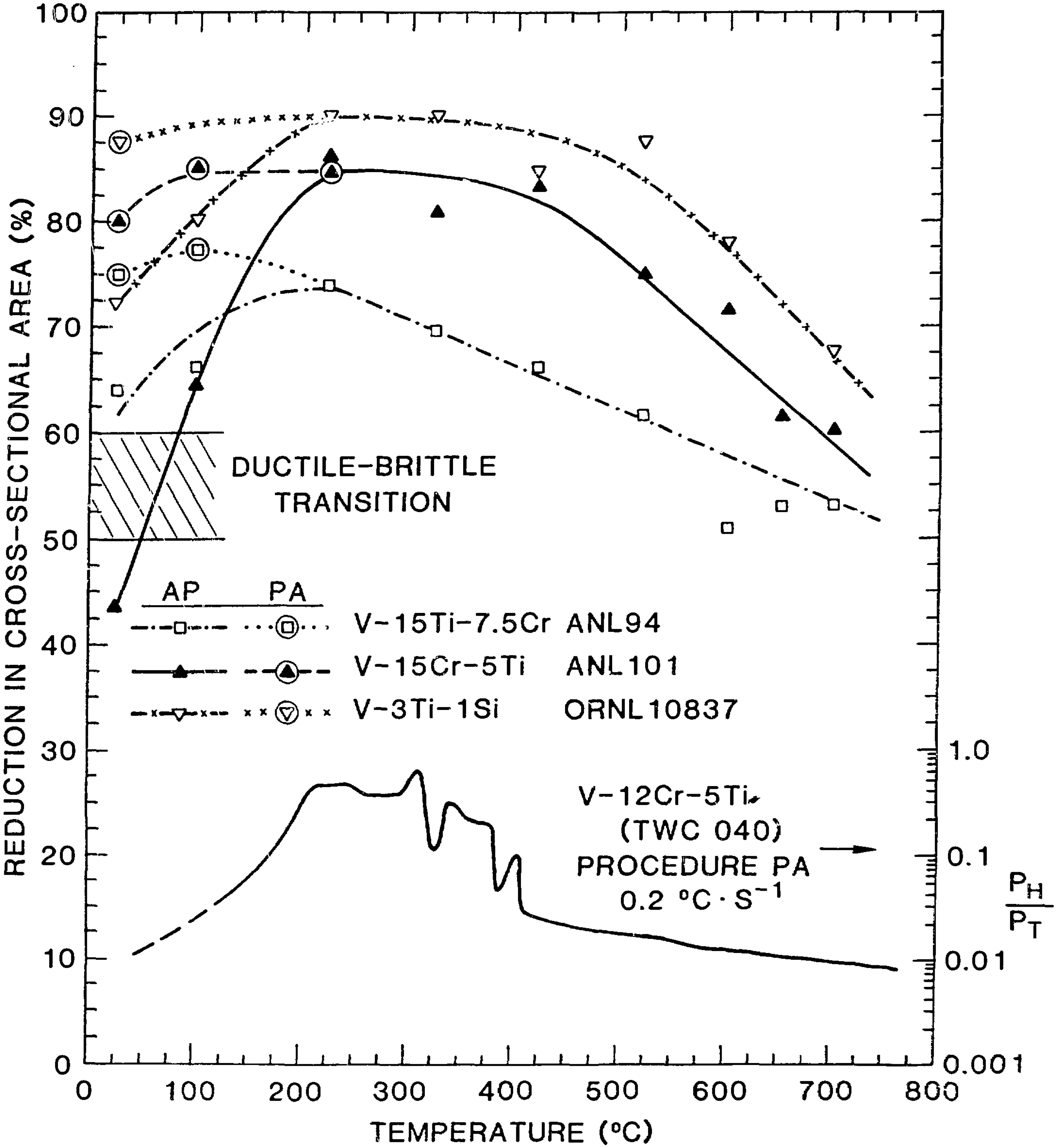

Fig. 4. Temperature Dependence of the Reduction in Cross-sectional Area for Vanadium Alloys.