Literature Review of Fracture Mechanics–based Experimental Data for Internal Hydrogen-assisted Cracking of Vanadium-modified 2¼Cr-1Mo Steel

API TECHNICAL REPORT 934-F, PART 2
FIRST EDITION, AUGUST 2017

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Downstream Segment

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Executive Summary

Literature data collectively suggest that V-modified 2¼Cr-1Mo base plate and weld metal each exhibit improved resistance to Internal Hydrogen-assisted Cracking (IHAC) compared to modern 2¼Cr-1Mo steel, where each steel is typified by modern-low J-factor or low X_B, and thus minimal temper embrittlement. IHAC in petrochemical applications typically involves H charging of the steel during elevated temperature exposure in high-pressure H_2, followed by stressing at near-ambient temperatures. For V-modified Cr-Mo, the slow-rising-displacement threshold stress intensity (K_{IH}) is a significant fraction of the H-free plane strain fracture toughness; however, V-modified steels are not immune to IHAC for this conservatively aggressive loading mode. This good IHAC resistance is attributed to beneficial H interaction with nano-scale vanadium carbide trap sites, but the details of this complex H trapping behavior are neither quantified nor fully understood.

No single study of IHAC in 2¼Cr-1Mo-0.25V base plate or weld metal is sufficiently comprehensive and quantitative to support fracture-mechanics modeling of pressure vessel fitness for service or a minimum pressurization temperature. Uncertainties in the existing data base are associated with:

a) the challenge in detecting K_{IH} in the midst of substantial crack tip plastic deformation;

b) slow crack growth rates typical of reversible H-trap interaction;

c) the lack of experimental results for very slow loading rates, weld metal, and stressing temperatures from -25 °C to 200 °C;

d) demonstration of H retention during an IHAC experiment;

e) lack of understanding of the fundamental mechanism by which H trapping at V_XC_Y precipitates reduces susceptibility to IHAC; and

f) the effect of low-temperature H charging.

All K_{IH} data for conventional and V-modified Cr-Mo steel reflect H charging followed by rising-displacement loading in moist air. A novel-deleterious interaction of IHAC and Hydrogen Environment-assisted Cracking (HEAC) for stressing of H precharged specimens in H_2 may occur, but is not supported by sufficient published data for Cr-Mo and Cr-Mo-V steels. H precharging, with stressing in moist air, or stressing in high-pressure H_2, without H precharging, are both capable of producing substantial H embrittlement in conventional Cr-Mo steel. Preliminary data show that V-modified Cr-Mo (without H precharging) is susceptible to HEAC in high-pressure H_2, conforming with the behavior of other low- to moderate-strength alloy steels, and apparently less affected by H trapping at vanadium carbides. K_{IH} values for uncharged 2¼Cr-1Mo-0.25V base plate stressed in high-pressure H_2 are substantially less than the thresholds typical of IHAC. The HEAC susceptibility of V-modified Cr-Mo should be considered in pressure vessel fitness-for-service modeling.

The ongoing laboratory study, commissioned by API Task Group 934F on Heavy Wall Pressure Vessel MPT, is addressing these uncertainties for 2¼Cr-1Mo-0.25V weld metal and base plate. Literature results obtained after about 2002 are sufficiently accurate and detailed to provide important comparisons with these newly emerging laboratory data. The result will be a strong characterization of the IHAC susceptibility of V-modified Cr-Mo steels.

Literature Review of Fracture Mechanics–based Experimental Data for Internal Hydrogen-assisted Cracking of Vanadium-modified $2^1/_4$Cr-1Mo Steel

1 Background

Between 1980 and 1990, researchers in Japan, France, and the United States determined that bainitic 2¼Cr-1Mo steels (UNS K21590) exhibited unexpectedly low-threshold K stress intensity levels (K_{IH}) for the onset of Internal Hydrogen Assisted Cracking (IHAC). Much of this work was sponsored by the American Petroleum Institute and the Materials Properties Council (MPC). Such cracking was observed for compact tension specimens that were fatigue precracked, precharged with atomic hydrogen (H) through elevated temperature exposure in high pressure H_2, and stressed under slow-rising crack mouth opening displacement (CMOD). The threshold for IHAC under static displacement loading was very high, as is expected for this moderate-strength steel.

While this class of Cr-Mo pressure vessel steels is clearly susceptible to significant IHAC under these conditions, initial data were uncertain for several reasons:

— lack of a standardized experimental method;

— detection of the onset of crack growth during rising CMOD was not rigorous;

— plasticity during loading complicated crack-growth detection and elastic stress intensity factor analysis;

— variables including actual loading rate in terms of dK/dt and H loss during testing were either not controlled or not reported;

— high variability in K_{IH} measurement both for a single laboratory and across groups;

— the mechanism for the deleterious effect of rising CMOD on IHAC was not elucidated; and

— much of this work was presented as hard copy associated with API-MPC committee presentations, or in conference proceedings, and was not published in peer-reviewed journals.

Between 1994 and 2000, a joint industry program (JIP) was conducted, including five laboratories in Japan, France, and the United States, to develop a rigorous-standard test method for measurement of K_{IH}, as well as crack-growth rate (da/dt) versus elastic-plastic stress intensity (K_J) and the threshold for the arrest of IHAC (K_{TH}) [1,2]. This method was then applied to develop a strong data base for IHAC of 12 heats of 2¼Cr-1Mo base plate and weld metal, with impurity composition (and degree of temper embrittlement) being a primary variable, as well as retained H concentration and applied dK/dt [2]. Figure 1 provides a collection of results from this work, plotted as K_{IH} versus total-dissolved H concentration for several low-impurity (J-factor < 100 wt pct) and thus low-Fracture Appearance Transition Temperature (FATT) (FATT < -28°C) heats of 2¼Cr-1Mo base plate and weld metal [3]. All experiments were conducted at 23 °C, with 25.4 mm-thick standard compact tension specimens precharged in H_2 at several combinations of elevated temperature and H_2 pressure. Results for the high-purity Phase I steels are shown by filled diamonds with the associated trend line [3]. Open and filled circles and triangles represent older literature data for a 2¼Cr-1Mo base plate that was not temper embrittled (J and FATT were not published, but are very likely low). The ultimate tensile strength of these steels was between 585 MPa and 605 MPa. The solid-to-dashed line shows the Phase I REACT software algorithm used to describe the H concentration dependence of K_{IH} for high-purity/low-FATT steels [2].

The severity of IHAC for this class of Cr-Mo steels is apparent in Figure 1, considering that the plane strain fracture toughness for this class of steels is of order 300 MPa√m at 23 °C. From 2002 through 2008, Phase II of this JIP centered on measurement and modeling of the effect of temperature on IHAC in 2¼Cr-1Mo base plate and weld metal, and a fracture mechanics–based method was developed to predict the minimum pressurization temperature based on IHAC under rising displacement [3,4]. This analysis augments determination of MPT governed by other important failure modes, including H-sensitive unstable fracture [5-7].

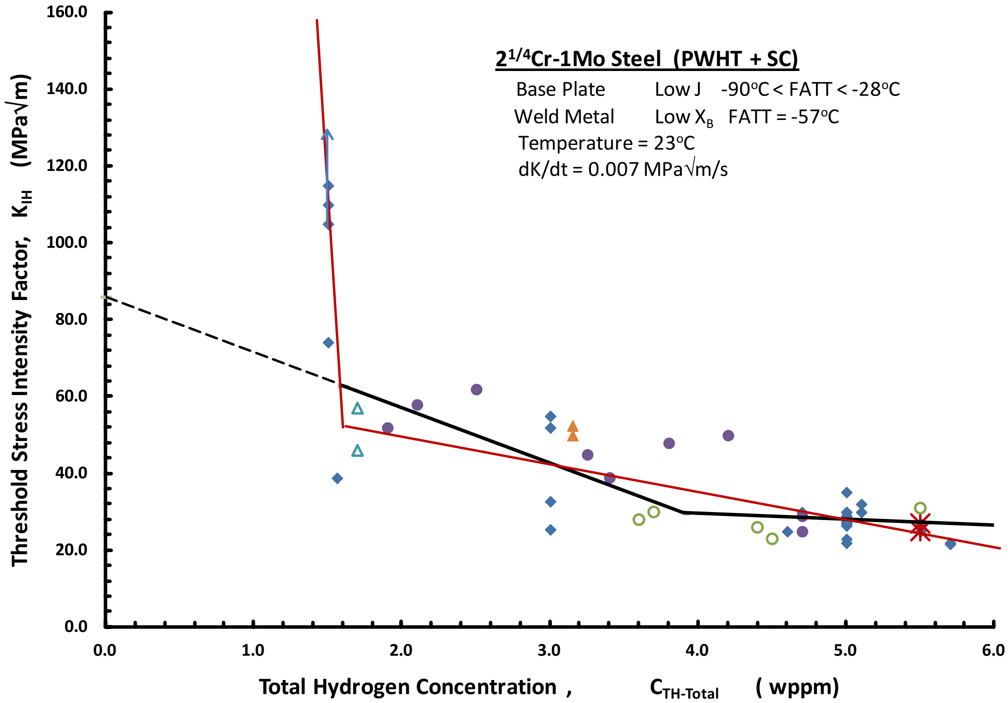


Figure 1 (after Gangloff [3])

In the early 1980s, in parallel with this focus on IHAC of Cr-Mo, vanadium-modified Cr-Mo steels were developed for heavy wall hydrocracking reactor applications [8,9]. The microstructure was almost always bainitic. The presence of homogeneously precipitated, nano-scale vanadium carbides (VC and V_4C_3) was hypothesized to greatly reduce the mobility of H through strong-reversible or even irreversible trapping, with the result being reduced susceptibility to IHAC. Fracture mechanics–based data were presented to characterize the IHAC performance of two primary compositions: 2¼Cr-1Mo-0.25V-0.035Nb-0.13C (UNS K31835, minimum yield strength (σ_{YS}) = 415 MPa, and minimum ultimate tensile strength (σ_{UTS}) = 585 MPa) and 3Cr-1Mo-0.25V-0.035Nb-0.13C-(Ti,B) (UNS K31830, minimum σ_{YS} = 415 MPa and minimum σ_{UTS} = 585 MPa).

These V-modified steels exhibited better resistance to IHAC under slow-rising CMOD compared to conventional Cr-Mo steel. However, these data are not sufficient to quantify the IHAC resistance of these steels because:

a) the issues noted above for 2¼Cr-1Mo are equally relevant to V-modified steels;

b) reduced H mobility likely reduces da/dt, which further complicates time-dependent experimental measurements relevant to long-term IHAC behavior;

c) the temperature dependence of IHAC depends on H trapping and may differ for the V-modified grades, and was not reported; and

d) small laboratory heats were used for a portion of the early laboratory experiments.

Given these uncertainties, API Task Group 934-F on Heavy Wall Pressure Vessel MPT commissioned a laboratory study of the IHAC resistance of modern 2¼Cr-1Mo-0.25V-0.035Nb-0.13C. The objective of this program is to employ the JIP-developed standard method to quantify the IHAC resistance of precracked specimens of modern 2¼Cr-1Mo-¼V base plate and weld metal. Particular emphasis is placed on detection of the onset of crack growth and state-of-the-art elastic-plastic analysis of K_J, as well as the primary variables of temperature and loading rate. The technical plan for this work was detailed in a report to the task group [10].

This report documents a critical assessment of the existing literature on IHAC of V-modified Cr-Mo steels for use in interpreting the results of the present laboratory work (reported in TR 934-F, Part 3, *Subcritical Cracking of Modern 2¼Cr-1Mo-¼V Steel due to Dissolved Hydrogen and H_2 Environment*) so as to establish a definitive characterization of the H cracking resistance of this steel class. Since these modern Cr-Mo-V steels are of relatively high purity, and thus retain a low Fracture Appearance Transition Temperature (FATT) after laboratory simulation of in-service temper embrittlement, the data base for 2¼Cr-1Mo shown in Figure 1 provides a context for assessment of the IHAC performance of V-modified grades. Hydrogen cracking of less pure V-modified Cr-Mo steels was not considered in this review. The content that follows is chronologically organized into initial and more modern works, as justified by improvement in test execution, data analysis and reporting, as well as the evolution from laboratory to commercial scale heats of Cr-Mo-V.

2 Results

2.1 Initial Experimentation; 1985–2000

In 1993, Ishiguro and co-workers published a substantial data set that previously had appeared in a variety of committee reports over the late 1980s. It described the IHAC resistance of bainitic 3.06Cr-1.0Mo-0.29V-0.14C-0.03Ti-0.0.002B [8,9]. This work was sponsored by API and MPC. The product form was a commercial 450 mm-thick forged shell. The J-factor for this V-modified steel is low—35 to about 100 wt pct—but uncertain since the Sn content of this steel was not published. The 40 ft-lb Charpy impact energy transition temperature was low (-40 °C) for the primary temper of interest (705 °C for 8 h), but K_{IC} for the forging at 23 °C was not reported. A laboratory scale heat of this composition and temper was characterized by K_{IC} at 23 °C of 200 MPa√m. Compact tension specimens were exposed in 19.6 MPa H_2 at 482 °C, fatigue precracked, and subjected to the slow-rising CMOD method. While not stated explicitly, dK/dt in these experiments is estimated to equal 12 MPa√m/h, prior to the onset of stable crack growth. (Analysis of compact tension specimen compliance, and an estimate of typical grip-loading rod compliance suggests that dK/dt (MPa√m/s) = 160 dCMOD/dt (mm/s) = 80(grip-displacement rate in mm/s) [2].) The K_{IH} is based on deviation of P versus CMOD from that typical of a H-free compact tension specimen. The dependence of K_{IH} on σ_{UTS} is shown in Figure 2 (1 kgf/mm^2 = 9.81 MPa), and on total-dissolved H concentration in Figure 3. Tempering temperatures were not specified for the IHAC results in Figures 2 and 3. However, the following data for the V-modified composition were provided in the same paper:

690 °C 40 h	σ_{YS} = 70.7 kg/mm^2	σ_{UTS} = 90.8 kg/mm^2
690 °C 4 h	σ_{YS} = 82.2 kg/mm^2	σ_{UTS} = 99.6 kg/mm^2
670 °C 4 h	σ_{YS} = 96.7 kg/mm^2	σ_{UTS} = 111.0 kg/mm^2
650 °C 10 h	σ_{YS} = 112.6 kg/mm^2	σ_{UTS} = 127.7 kg/mm^2

For comparison, K_{IH} data from these authors are presented in Figures 2 and 3 for a conventional, high purity, bainitic 2¼Cr-1Mo steel base plate (J = 38 to 100 wt pct with Sn not reported; 40 ft-lb Charpy transition temperature = -92 °C; and K_{IC} at 23 °C for a laboratory scale heat = 200 MPa√m). These conventional and V-modified bainitic steels are similar in terms of purity and fracture toughness, without precharged H, so the primary difference in IHAC resistance must be reasonably related to V addition and the resulting microstructure.

The data in Figure 2 show that the V-modified steel is susceptible to IHAC, as evidenced by the fact that K_{IH} is less than K_{IC} for the H-free steel, which is about 200 MPa√m at 23 °C. However, K_{TH} is two to three times higher than values measured for conventional 2¼Cr-1Mo at the same ultimate tensile strength, but with somewhat higher total-dissolved H content. (Note that the K_{IH} values for the 2¼Cr-1Mo steels in Figures 2 and 3 are in good agreement with the modern JIP-based data set plotted in Figure 1 for Cr-Mo steel with an ultimate tensile strength of 585-605 MPa or 60 to 62 kgf/mm^2). This comparison validates the experiments used to develop the data in Figures 2 and 3, at least for the conventional steel.

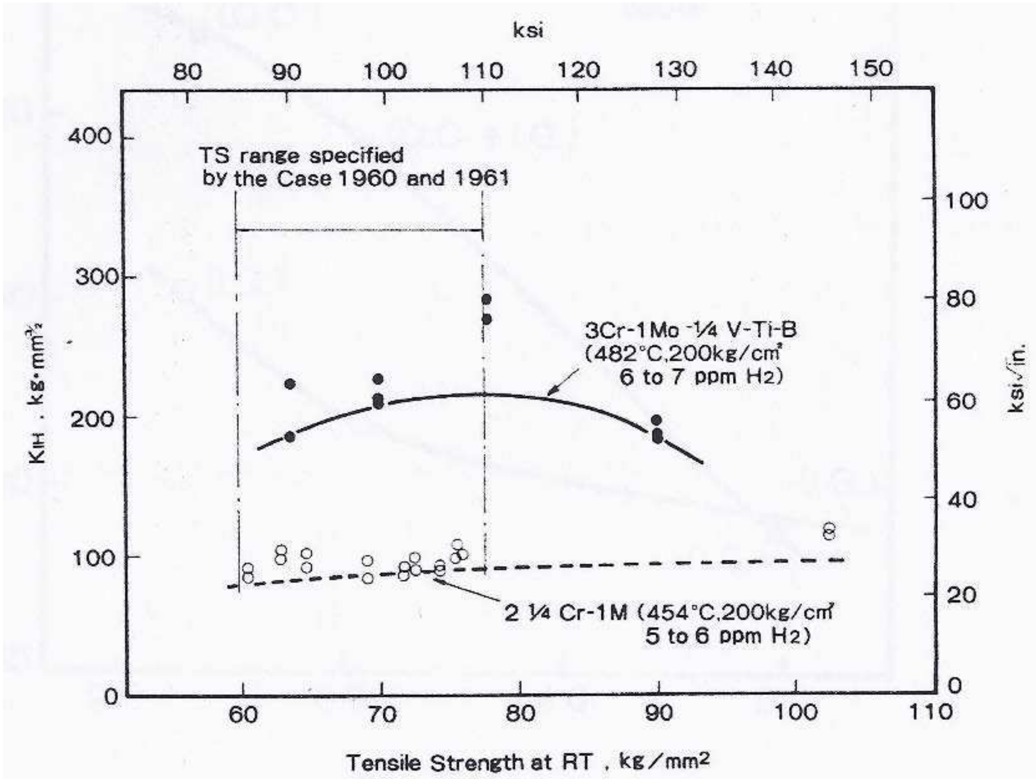


Figure 2 (after Ishiguro, et al. [8,9])

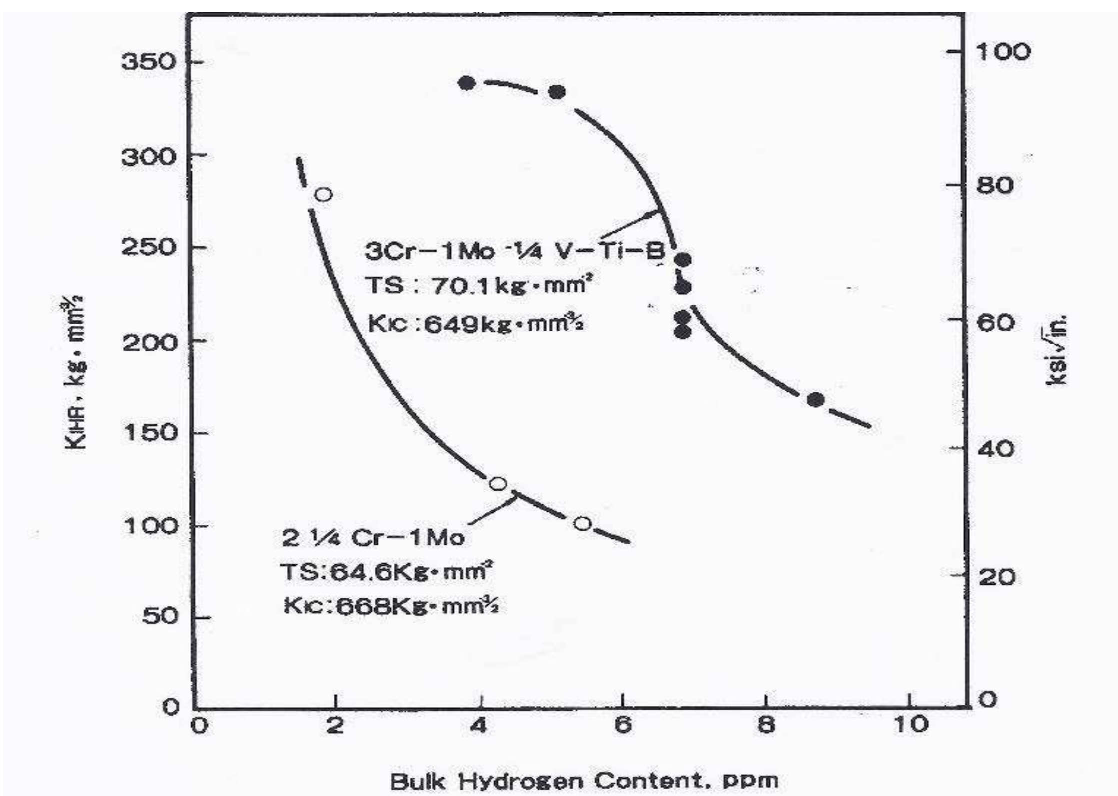


Figure 3 (after Ishiguro, et al. [8,9])

Figure 4, presented in an untitled handout apparently by the authors of Figures 2 and 3, suggests that increasing bulk H concentration in Figure 3 was achieved by increasing the temperature of high-pressure H_2 exposure for each steel. Neither tempering temperature nor ultimate tensile strength were reported for the steels in Figure 4. The strain rate in the legend of Figure 4 is actually the cross-head speed and translates to an estimated dK/dt of 14 MPa√m/h. Several points are important. First, a larger-total H concentration is produced by the same exposure applied to the V-modified steel. Second, fracture mechanisms are noted in Figure 4. For the conventional steel, the IHAC mode transitions from transgranular (so-called quasi-cleavage, which may be associated with H cracking along or across bainite interfaces [4]) to intergranular (IG) as precharged H concentration increases. For the V-modified grade, cracking is initially by microvoid-based processes, then transitions to transgranular IHAC, and finally IG IHAC for the two highest H-charging temperatures (500 °C and 550 °C).

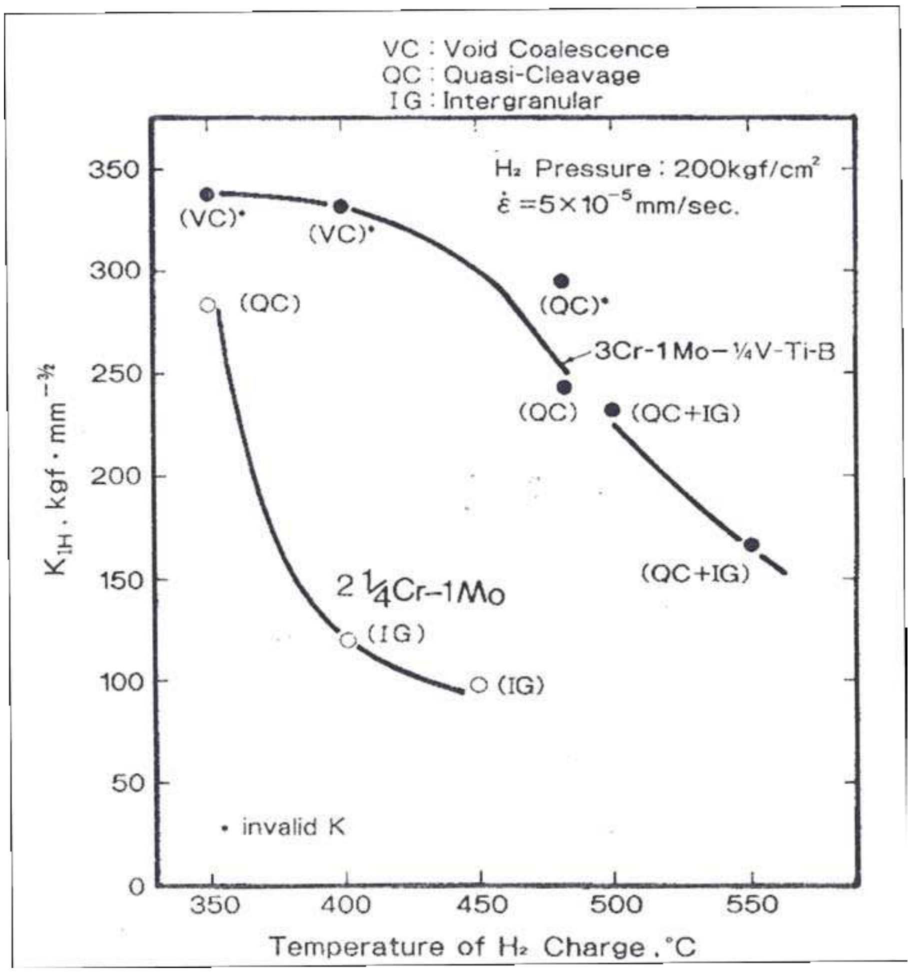


Figure 4 (apparently by Ishiguro et al. (about 1987) [8,9])

The microstructures of these two steels are characterized in Figure 5 [8,9]. Tempering at 690 °C produced the unique presence of VC in the V-modified steel, but no resolvable Mo_2C. Figure 6 shows that the amount of VC precipitate rises as the Cr content is reduced from 3 wt pct to 2¼ wt pct [8,9]. This lower Cr concentration has emerged as a focal point of more recent IHAC experimentation, including the work being conducted for the API task group. Given the possible difference in VC volume fraction, the data contained in Figures 2 through 4 provide guidance on the IHAC resistance of V-modified 2¼Cr-1M0 steel, but are not sufficient for a rigorous MPT and fracture mechanics fitness-for-service assessments. That said, if H trapping at VC precipitates is the basic reason for improved IHAC resistance, then a V-modified 2¼Cr composition should exhibit even better performance compared to that suggested by the comparison in Figure 2.

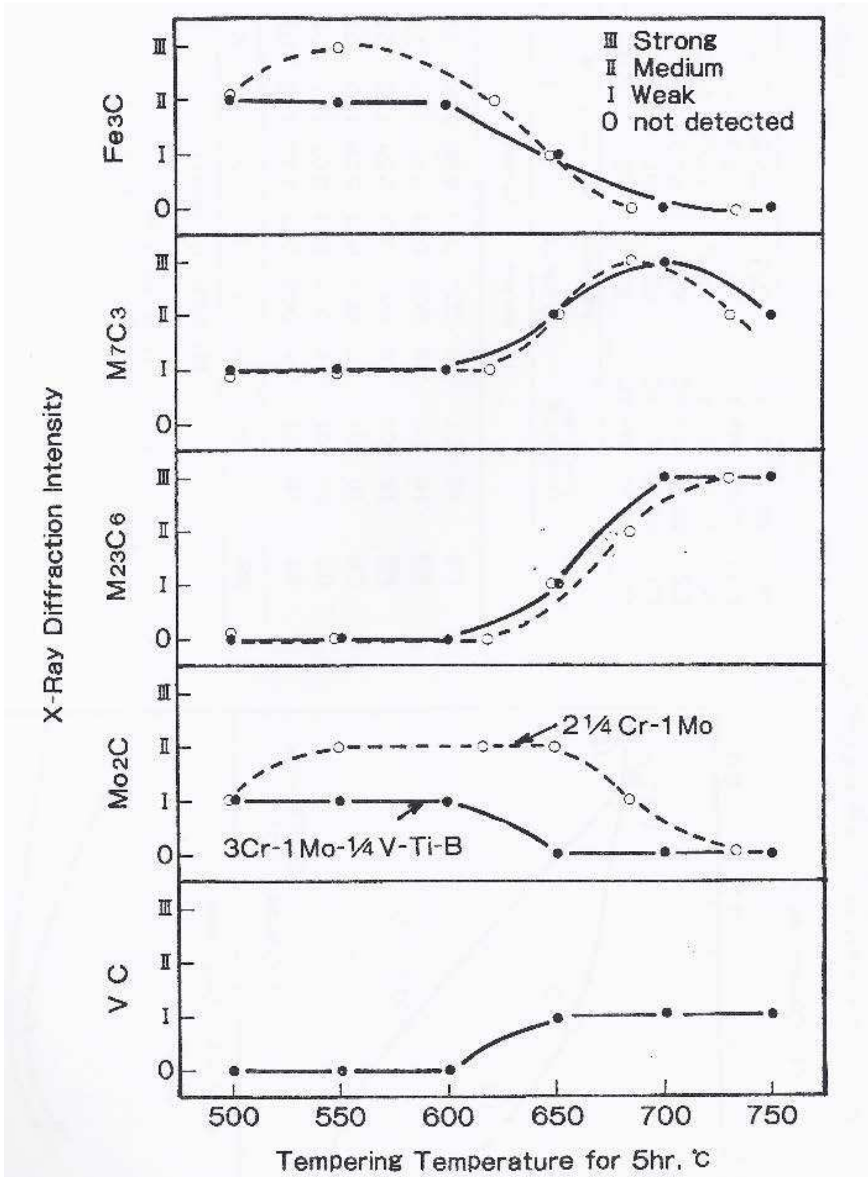


Figure 5 (after Ishiguro, et al. [8,9])

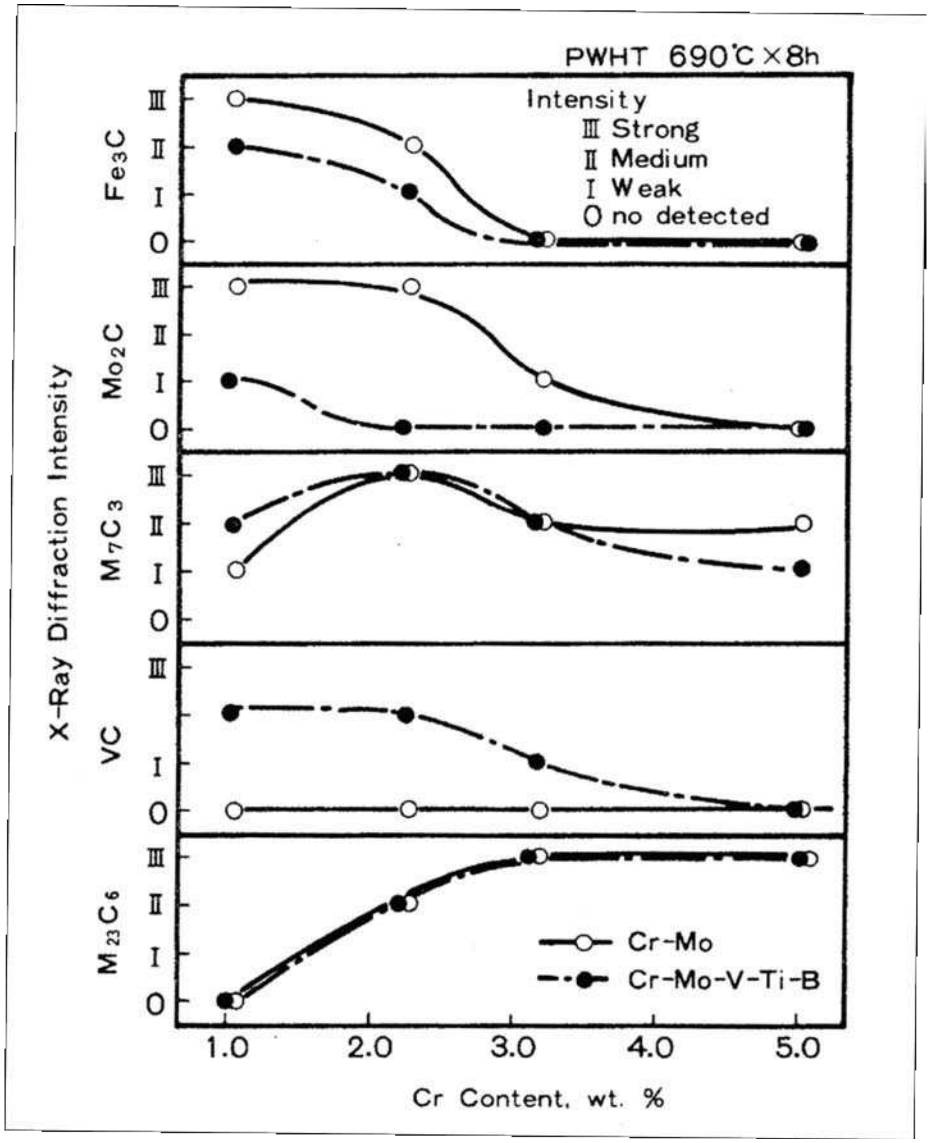


Figure 6 (after Ishiguro, et al. [8,9])

Ishiguro and co-workers presented extensive data showing strong differences in the trap-sensitive H diffusion behavior of V-modified steels compared with conventional Cr-Mo steels. Typical results are presented in Figures 7 and 8, which show the reduction in initial H concentration (from a 2.5-cm cube precharged at elevated temperature in H_2) plotted as a function of exposure time in moist air at various outgassing temperatures (Figure 7) and for various tempering temperatures with H egress at 23 °C (Figure 8, where C_o is the initial-total H content, C_t is the total-H content after exposure for time, t, and C_{720} is the total H content present after outgassing for 720 h.). These data suggest three important points:

1) H diffusion in the V-modified grade is slowed compared to that in conventional Cr-Mo for any outgassing temperature, attributed to H trapping at V_XC_Y.

2) H diffusion is measurable in 3Cr-1Mo-0.3V-Ti,B, suggesting that H trapping at V_XC_Y is reversible.

3) H diffusion and trapping in the V-modified composition depend on tempering temperature.

Speculatively, this strong temper dependence of H trapping in the V-modified grade suggests that the associated IHAC resistance could vary in a weld heat–affected zone of Cr-Mo-V steel in those locations where V_XC_Y coarsened at temperatures above a subsequent post-weld heat-treatment temperature.

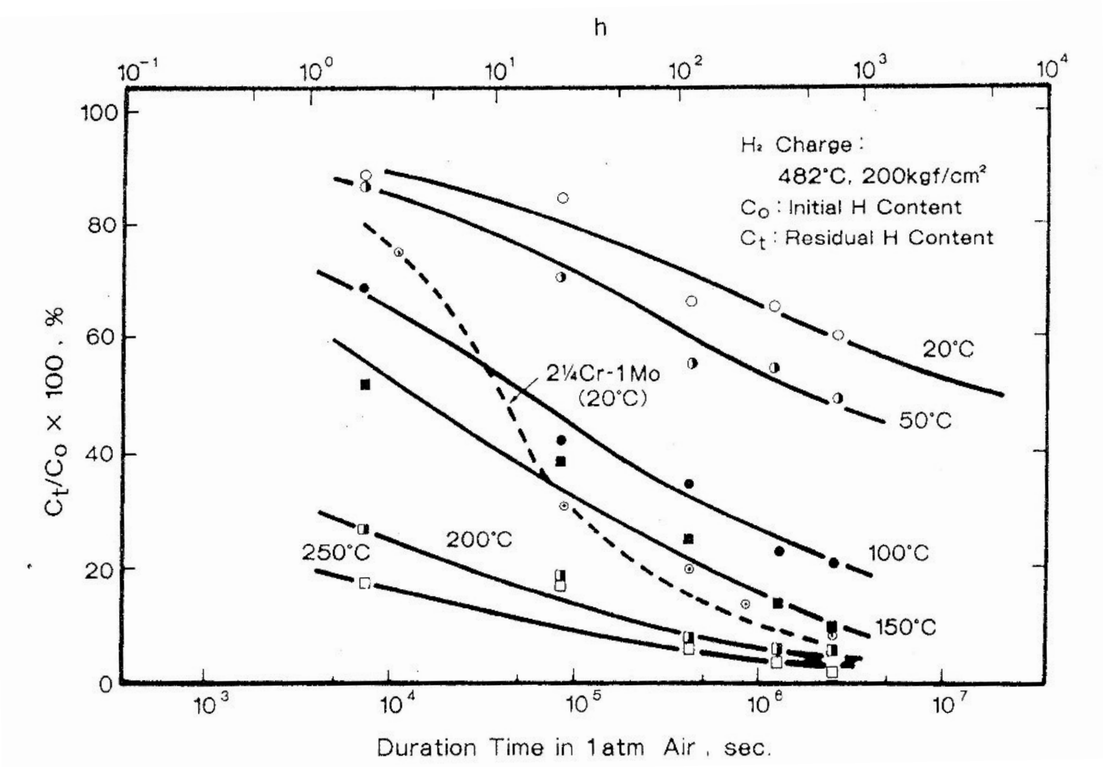
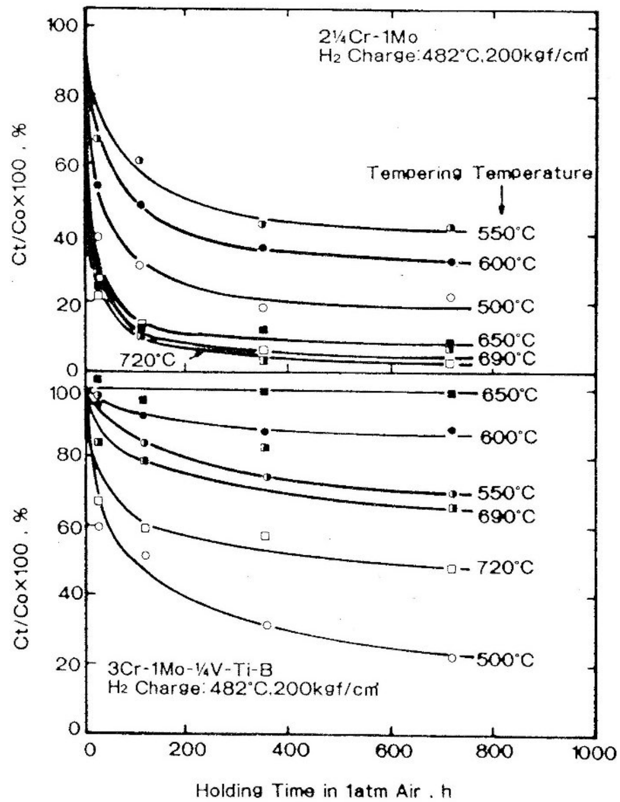


Figure 7 (after Ishiguro, et al. [8,9])



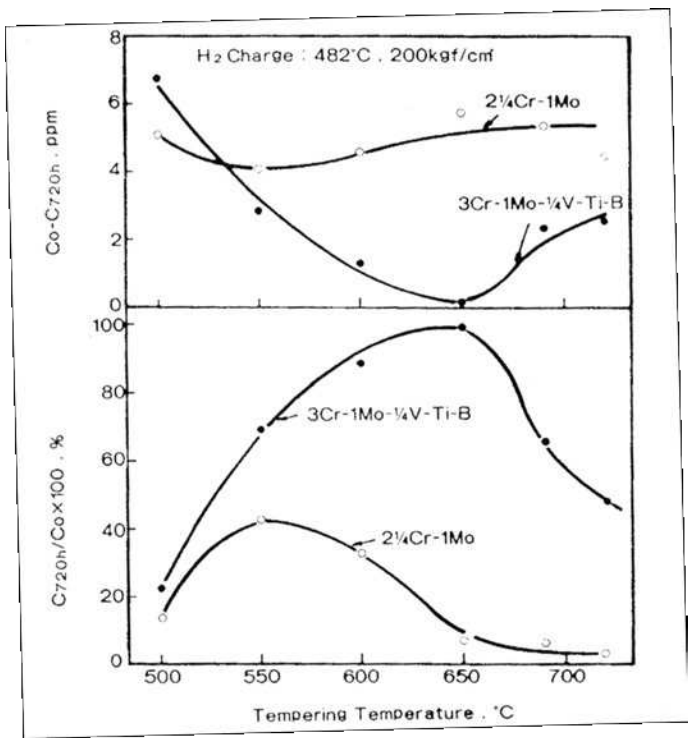


Figure 8 (apparently by Ishiguro et al. (about 1987) [8,9])

Figure 9 was presented, by these same authors and for this Cr-Mo-V-Ti-B steel, to a 1987 API-MPC meeting in Atlanta [8,9]. Susceptibility of this V-modified steel decreases to apparently zero as the testing temperature increases from 25 °C to 150 °C for the unspecified loading rate employed. Conventional Cr-Mo steel exhibits similar improvement in IHAC resistance as temperature rises above 100 °C [2,4].

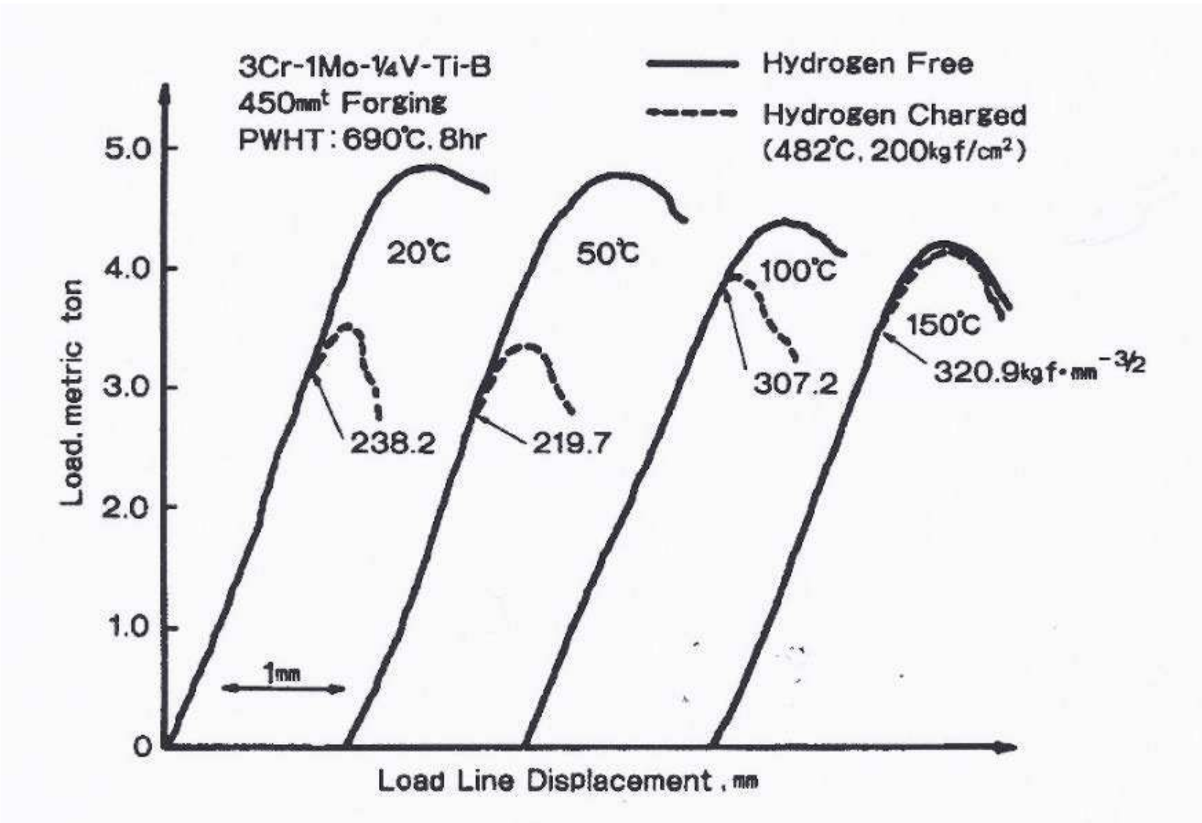


Figure 9 (after Ishiguro, et al. [8,9])

Beginning in the late 1980s, and continuing through about 1995, Coudreuse and co-workers published the results of experiments aimed at characterizing the IHAC of V-modified 2¼Cr-1Mo steel [11–13]. These results were affirmed in recent review papers [14–16]. The data in Figure 10 were replotted from a 1986 paper published by Sakai and co-workers in Japan [15]. The composition of each steel is nominally 2¼Cr-1Mo, each was tempered to yield a σ_{UTS} of about 690 MPa, which is relevant to the present study of IHAC in Cr-Mo-V, and the product form is base plate. All steels were H precharged at 450 °C in high-pressure H_2 (15 to 20 MPa). The parameter, F, is the percent reduction in uniaxial tensile reduction-in-area due to precharged H compared with the as-received case. Two points are notable:

1) IHAC is reduced as the V content rises;

2) The total and trapped H contents of this steel increase as V rises to 0.25 wt %. These various H concentrations were determined by outgassing experiments.

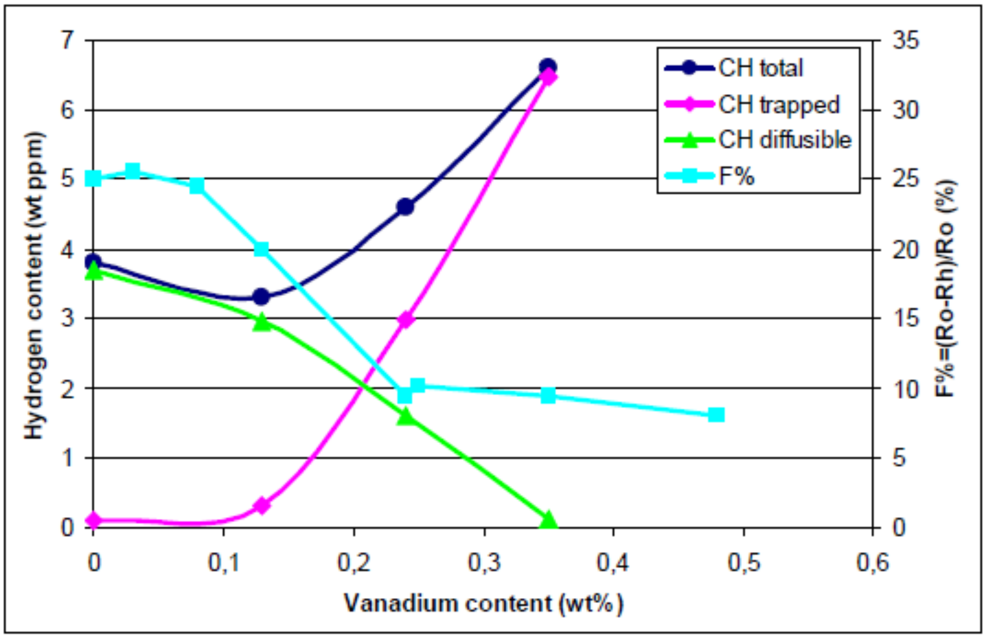


Figure 10 (data due to Sakai, Takagi, and Asami, 1986; after Pillot and Coudreuse [15])

Coudreuse and co-workers used the slow-rising CMOD experiment to measure K_{IH} for V-modified Cr-Mo steel, building on their extensive work with IHAC in conventional 2¼Cr-1Mo [2]. Initial experiments focused on a laboratory heat of 2.3Cr-1.0Mo-0.25V-0.13C tempered at either 685 °C for 7 h (σ_{YS} = 580 MPa and σ_{UTS} = 680 MPa) or at 700 °C for 7 h (σ_{YS} = 560 MPa and σ_{UTS} = 660 MPa) [11]. Compact tension specimens (25.4 mm thick) were H charged at 450 °C in H_2 at several high pressures, which produced total H concentrations between 3.4 and 6.8 wppm. They were then subjected to slow-rising CMOD at an unspecified rate and at 23 °C. Plasticity was significant, and there was no resolvable difference in the load versus CMOD curves for as-received and H precharged specimen, suggesting a strong resistance to IHAC for this V-modified steel. Fracture surfaces where similar, with and without H precharging. The H-free steel exhibited somewhat brittle behavior, perhaps not typical of a commercial heat, and this initial result is not definitive.

In this same time frame, Coudreuse et al. published important results on the IHAC resistance of 2.2Cr-1.0Mo-0.27V-0.14C base plate after H charging in either high-pressure H_2 (450 °C, 15 MPa H_2, 24 h) or in an ambient temperature acidified and H_2S-saturated chloride (NACE) solution [12]. Steels were tempered at 690 °C for 25 h (A; σ_{YS} = 501 MPa and σ_{UTS} = 613 MPa) or at 680 °C for 9 h (B, σ_{YS} = 627 MPa and σ_{UTS} = 723 MPa). Compact tension specimens (25.4 mm thick and side-grooved) were subjected to slow-rising CMOD loading at a CMOD rate of 0.005 mm/min, which translates to an estimated dK/dt of 48 MPa√m/h. All IHAC experiments were conducted at 23 °C. K_{IH} was calculated from J_o, assigned to the first deviation in the load versus CMOD plot for the H precharged specimen, and compared with the load versus CMOD response of as-received steel. It was assumed that this deviation is due to the onset of crack extension, which is reasonable provided that the H-charged and H-free specimens exhibit the same compliance and level of plasticity. A second measure of IHAC, K_J, was taken from $J_{0.2}$ established at a crack extension of 0.2 mm. Presumably, such crack extension was obtained from an unloading compliance measurement, but this detail was not specified.

The results of these experiments are tabulated in Figure 11, including data for conventional Cr-Mo base plate.

Steel	H_2 charging	J_0 (kJ/m2)	$J_{0,2}$ (kJ/m2)	dJ/da (MPa)	K_J (MPa√m)	K_{IH} (MPa√m)	CTOD (mm)	H_2 (ppm)
2.25Cr-1Mo treat. A	ref.	464	590	631	329	/	0.71	/
	gaseous (1)	54	77	110	112	80	0.18	2.5
2.25Cr-1Mo treat. B	ref.	417	540	468	312	/	0.53	/
	gaseous	49	67	88	107	85	0.11	2.7
	NACE (2)	79	97	88	136	80	0.13	2.5
2.25Cr-1Mo treat. C	ref.	425	490	322	315	/	0.37	/
	gaseous	60	74	71	118	90	0.1	2.9
2.25Cr-1Mo-V treat. A	ref.	265	390	495	249	/	0.43	/
	gaseous	200	270	170	216	>150	0.22	4.2
	NACE	17	39	52	63	55	0.07	4.4
2.25Cr-1Mo-V treat. B	ref.	231	257	124	232	/	0.25	/
	gaseous	221	248	131	227	>150	0.18	4.4
A516 gde70	ref.	163	301	492	195	/	>0.85	/
	NACE	11	26	75	51	50	0.06	1.1
SE 500	ref.	378	615	791	297	/	>0.85	/
	NACE	35	63	133	90	80	0.13	1.1

Figure 11 (after Coudreuse et al. [12])

Figures 11 and 12 demonstrate that the threshold stress intensity for the onset of IHAC is much higher for the V-modified steel, which is precharged at elevated temperature in H_2, compared to conventional Cr-Mo base plate. For the Cr-Mo base plate, both K_{IH} and the J-R curve for the H-charged case are substantially lowered compared to that characteristic of the V-modified steel. In fact, H cracking in the V-modified steel shows essentially identical first-initiation “toughnesses” with and without H charging. The J-R curve is lowered somewhat by H precharging for the 2¼Cr-1Mo-0.25V steel. This enhanced threshold is in spite of the fact that the measured-bulk H concentration for this constant charging condition was almost twice as high for 2¼Cr-1Mo-0.25V, consistent with the data shown in Figure 4. This result is tempered by the fact that a single, relatively rapid dK/dt was employed and the precharged H concentration was moderate (~4 wppm).

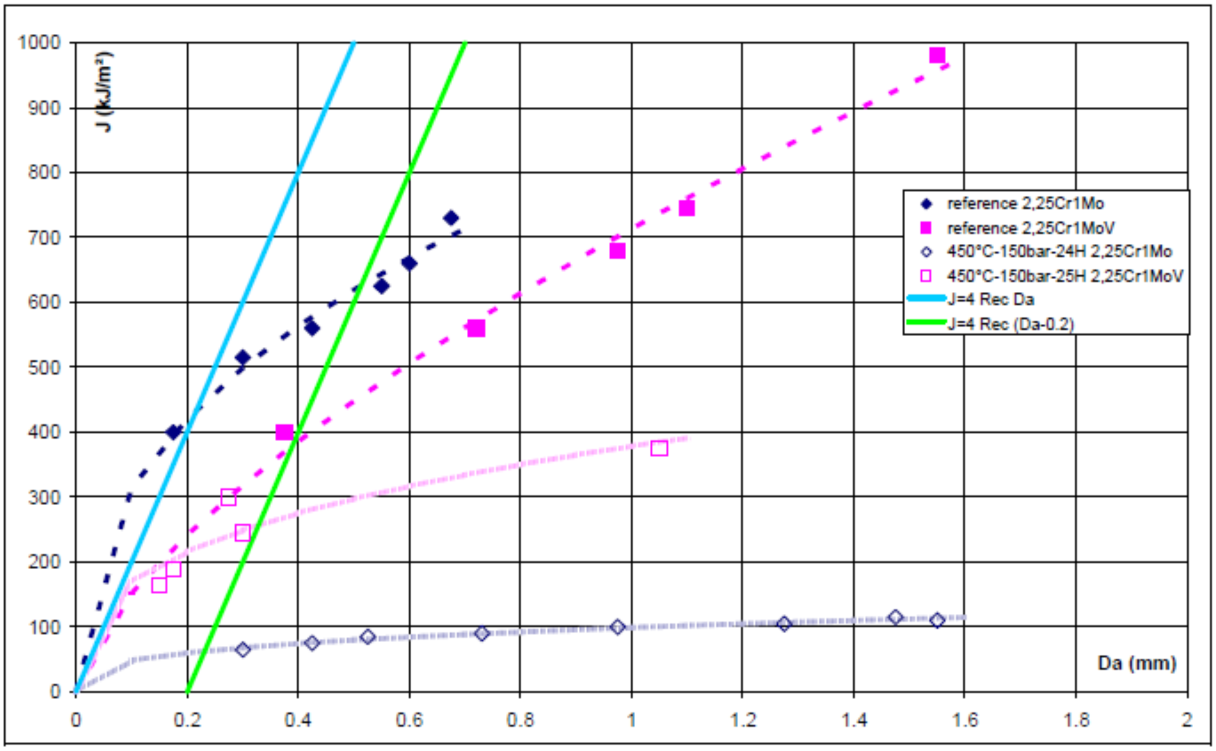


Figure 12 (after Coudreuse et al. [12]; replotted in [16])

Coudreuse et al. examined the effect of the loading rate on IHAC in Cr-Mo-V steel, with the results presented in Figure 13 for crack mouth opening displacement rates of 0.005, 0.05, and 0.5 mm/min for the V-modified steel [12]. These three grip-displacement rates correspond to initial-applied dK/dt levels of 48, 480, and 4,800 MPa√m/h (0.013, 0.13, and 1.3 MPa√m/s). The extensive results shown in Figure 14 for conventional Cr-Mo steel establish that K_{IH} is loading rate–independent for dK/dt up to 0.4 MPa√m/s and perhaps higher (equivalent to an approximate dCMOD/dt value of 0.15 mm/min shown by the vertical arrow in Figure 13) [2]. However, since the mobility of H in the V-modified steel is 100 or more times slower compared to Cr-Mo, the loading rates examined by Coudreuse et al. (Figure 13) may not be sufficiently slow to adequately characterize the IHAC resistance of this steel.

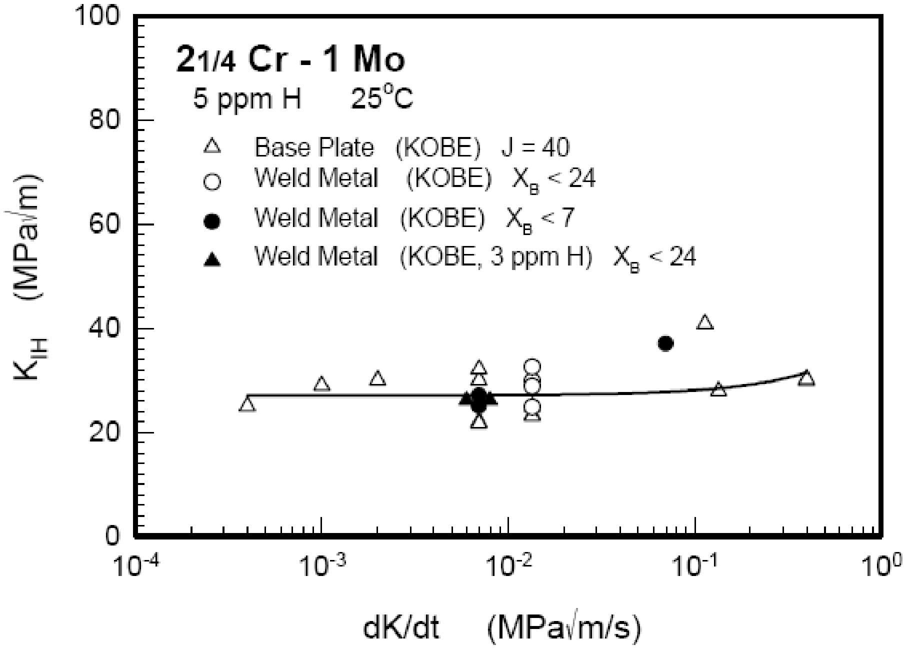


Figure 13 (after Coudreuse et al. [12]; replotted in [16])

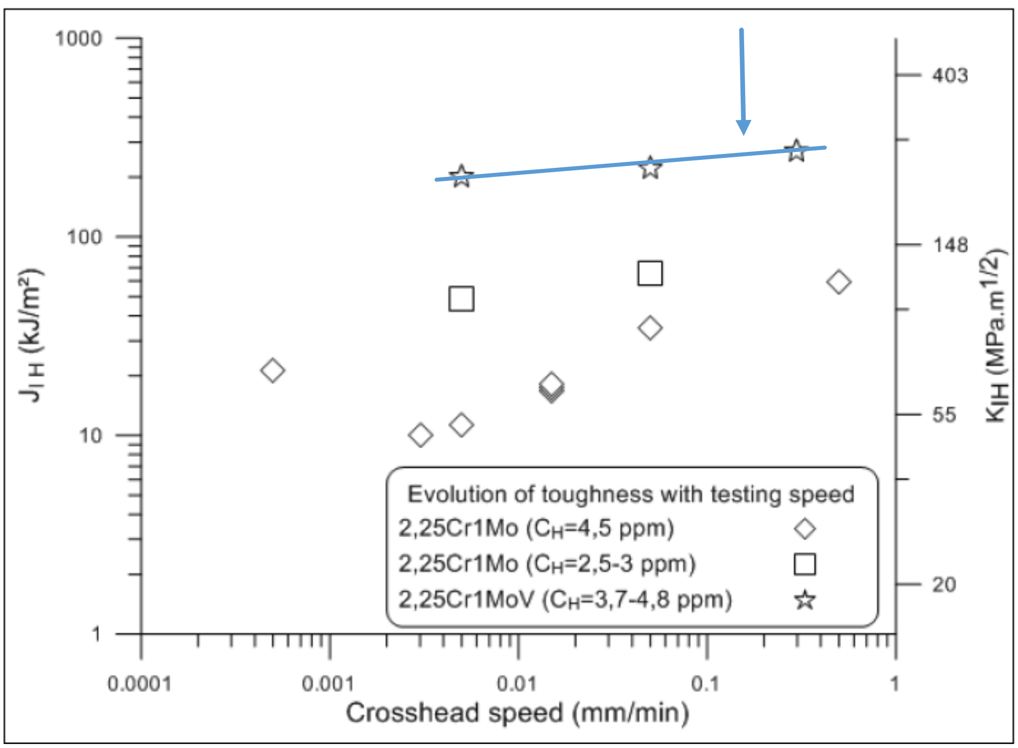


Figure 14 (after Gangloff [2])

This study by Coudreuse and co-workers is notable in that the effect of the H charging source and charging temperature was investigated [12,15,16]. As tabulated in Figure 11, and plotted in Figures 15 and 16, H precharging in NACE solution at 23 °C promoted IHAC in this heat of 2¼Cr-1Mo-0.25 V base plate. The K_{IH} was above 150 MPa√m, and K_J was 220 MPa√m and 230 MPa√m, for the experiments conducted with H_2 precharged specimens of tempers A and B (Figure 15). In contrast, these IHAC thresholds were K_{IH} = 52 MPa√m and K_J = 58 MPa√m for the NACE precharged specimen of the A temper.

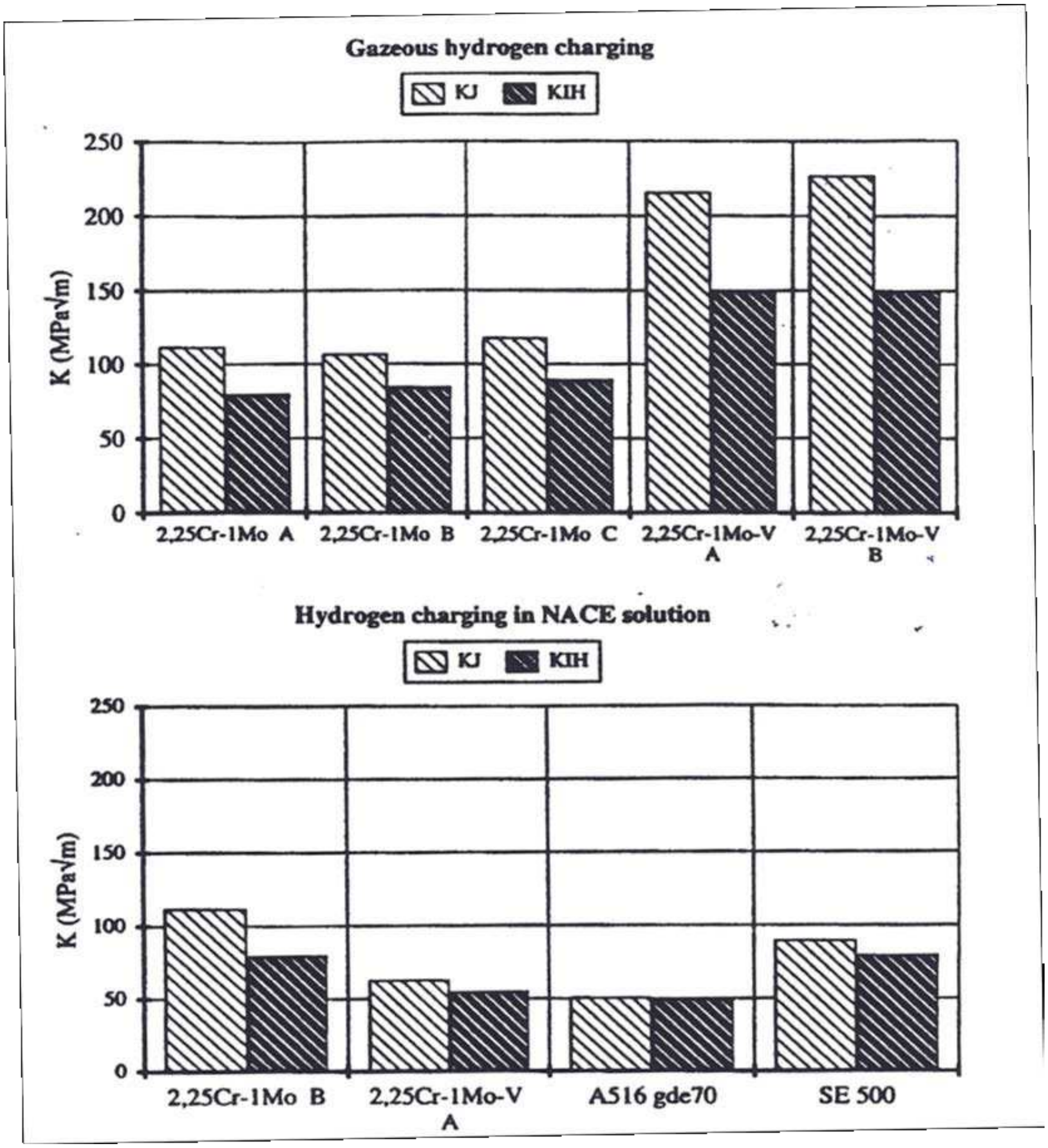


Figure 15 (after Coudreuse et al. [12])

Considering Figure 16, presumably, temper A is HT1 and temper B is HT2 in this replotted form of the Figure 15 data. However, the noted values of σ_{UTS} do not match when comparing A (σ_{UTS} = 613 MPa, Figure 15 [12]) and HT1 (σ_{UTS} = 650 MPa, Figure 16 [15]). The two J-based thresholds for IHAC in Figure 16 are equivalent to K_{IH} and K_J of 61 and 93 MPa√m for HT1 of the V-modified steel charged in NACE solution, compared with K_{IH} of 211 MPa√m and K_J of 245 MPa√m for HT1 charged in high-temperature H_2. The differences between the results in Figures 15 and 16 are not understood.

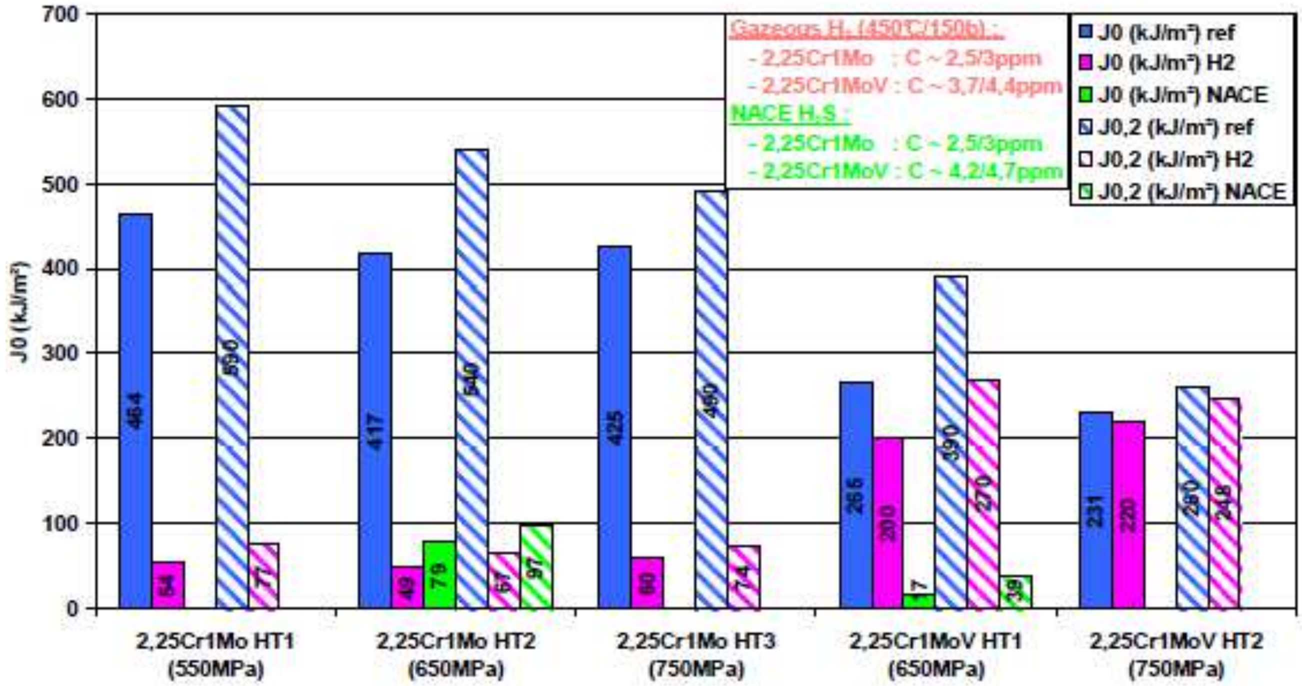


Figure 16 (after Coudreuse et al. [12]; replotted in [15])

Two points are notable regarding the effect of H-charging source (temperature) represented in Figures 15 and 16. First, the measured-bulk H concentrations for temper A (HT1) of the V-modified steel were 4.2 wppm from elevated temperature H_2 charging and 4.4 wppm from room temperature NACE charging [12], restated as 4.0–4.8 wppm for H_2 and 4.2–4.7 wppm for NACE charging [15]. Also notable is the point that essentially the same IHAC threshold parameters were measured for conventional Cr-Mo base plate, independent of the H charging source or temperature, as shown in Figure 15 [12,15]. Somewhat higher values of K_{IH} and K_J are reported for the Cr-Mo/NACE solution case shown in Figure 16 for HT2, compared with the values given in Figure 15 for temper B of the conventional 2¼Cr-1Mo steel. The origin of this plotting difference is not known.

Strong IHAC produced by room-temperature H precharging is potentially important for situations where V-modified Cr-Mo steel is exposed to high-H fugacity charging conditions during pressure vessel shutdown. This result must be verified, given that only a single result is reported for 2¼Cr-1Mo-0.25V base plate in Figures 15 and 16. Moreover, a uniquely damaging effect of H charging temperature was not observed for uniaxial tensile specimens of either conventional or V-modified 2¼Cr-1Mo steels, when percentage reduction in ductility (F parameter) was plotted versus the measure-bulk H concentration from several environments (Figure 17 [15]).

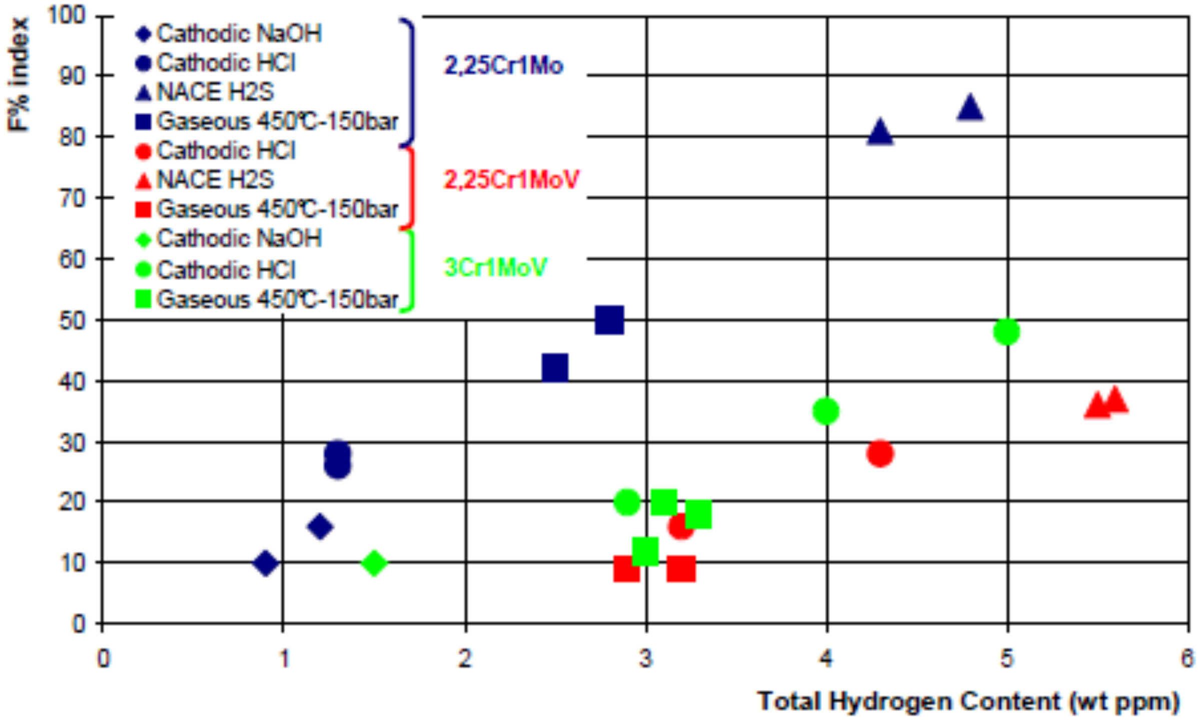


Figure 17 (after Pillot et al. [15])

An effect of H charging temperature on IHAC was interpreted as associated with different distributions of H between lattice sites, reversible trap sites, and irreversible trap sites [15]. The general hypothesis is that charging at 23 °C in the high-H fugacity NACE solution tends to saturate V_XC_Y trap sites, which is typical of "open system" H trapping [17] and which leads to substantial mobile H that enables IHAC. In contrast, charging at elevated temperature initially produces lattice soluble H in equilibrium with this open gas system, but such H partitions to V_XC_Y trap sites on cooling, characteristic of a "closed system" [17]. Closed-system H trapping is either strongly irreversible (if H enters the carbide lattice by virtue of sufficient thermal-activation energy) or strong-reversibly trapped to reduce the kinetics of IHAC or to preclude H repartition to the crack tip stress field. The precise explanation for this behavior requires quantitative values of H-trap binding energy for V_XC_Y, bainite interfaces, and the crack tip stress field, coupled with a formal analysis of the distribution of H trapping among multiple sites.

Evolving work by Pillot and co-workers demonstrates that H outgassing is substantially faster for specimens that are precharged at 23 °C, in either NACE solution or under cathodic polarization in H_2SO_4, compared to H egress after elevated-temperature H_2 charging. These results support the proposed effect of H charging condition on H trapping in trap-rich V-modified Cr-Mo steel [15]. Specific data are presented in Figure 18. The solid data points show H egress from 2¼Cr-Mo-0.25V, at 20 °C and after H charging in three different environments: (a) "20C" having been charged in 15 MPa H_2 at 450 °C, (b) "1 N H_2SO_4" for H charging in 1N sulfuric acid at 20 °C, and (c) and "H2S" for H charging in NACE solution at 20°C. The three sets of open points represent H egress from a separate heat of 2¼Cr-1Mo-0.25V, precharged in high pressure H_2 and permitted to outgas at 20 °C, 100 °C, and 200 °C. The very slow H diffusion at 20 °C is reproduced compared to the filled diamond results, and outgassing at 200 °C progressed at a rate very similar to that observed for conventional 2¼Cr-1Mo steel outgassed at 20 °C. Results for 3Cr-1Mo-0.25V steel were essentially identical to those shown for 2¼Cr-1Mo-0.25V in Figure 18. Interpretation of the cause of the different outgassing kinetics shown in Figure 18 is clouded by the possibility that the H distribution in the NACE charged, 25-mm-diameter cylindrical specimen was not uniform, owing to slow H uptake at 23 °C. Moreover, note that the very slow H egress suggested in Figure 18 is substantially less than that suggested by the data in Figure 7 for the 3Cr-1Mo-0.3V composition. Additional experiments are required to elucidate the effect of H charging condition on the V_XC_Y trap-sensitive diffusivity of H.

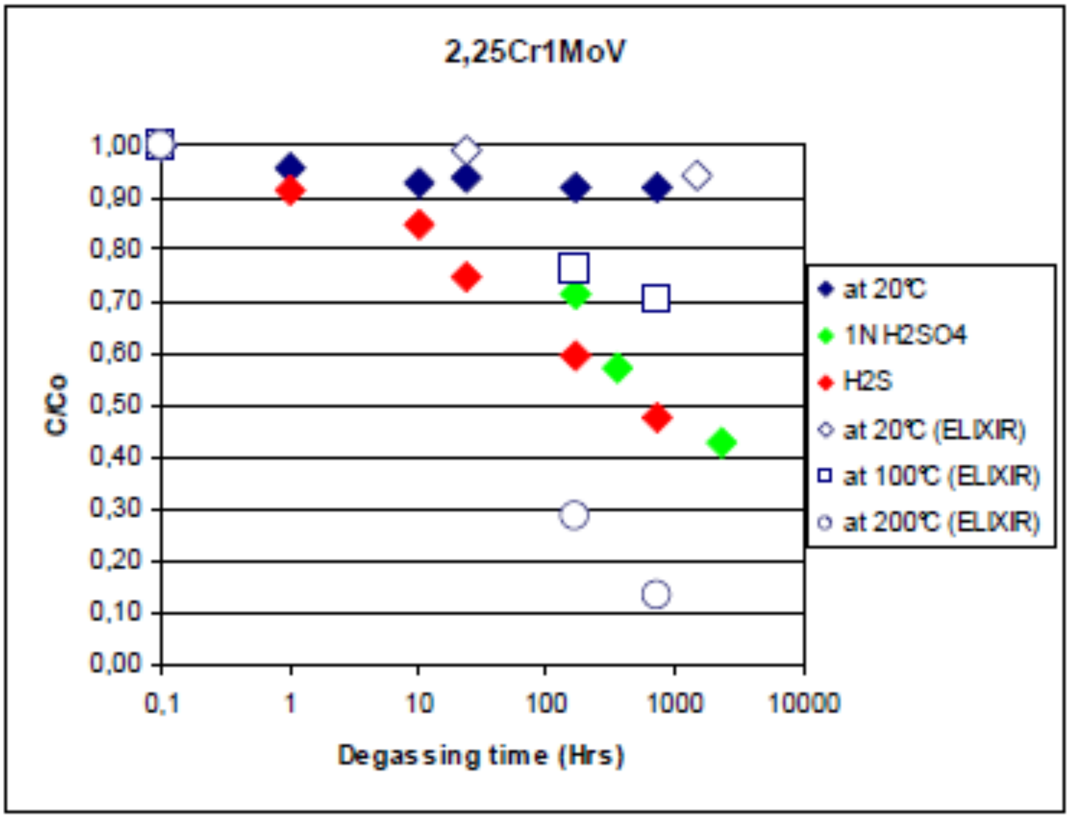


Figure 18 (after Pillot et al. [15])

2.2 Improved Experimental Characterization of IHAC; 2000–present

The results of IHAC experiments conducted after 2000 are considered to be particularly accurate and relevant due to improved measurements of subcritical crack growth, as well as refined calculations of stress intensity factor from elastic-plastic J-integral analysis.

In 2002, Wada and co-workers published data on the IHAC resistance of 2.45Cr-1.05Mo-0.29V-0.14C base metal (PWHT at 705 °C for 8 h) with FATT of -78 °C (J-factor = 38 wt pct) and σ_{UTS} of 663 MPa [18]. Side-grooved (25.4 mm thick CT specimens) were H charged at 480 °C in 20 MPa H_2 to produce a total-dissolved H concentration of 12 wppm. Charged specimens were subjected to slow-rising CMOD loading (dK/dt stated to equal 18 MPa√m/h). The amount of H remaining after a 120 h fracture experiment at 23 °C was reported to be 9 wppm. K_{IH} was established from multiple-interrupted J versus stable crack extension (J-R curve) experiments.

Example J-R curves for several loading temperatures are presented in Figure 19, and the temperature dependence of K_{IH} is given in Figure 20. The experiments used to produce these results were improved over earlier data, perhaps due to the participation of these authors in the JIP program, which developed a reasonably standardized fracture-mechanics test method and elucidated the effects of important variables on K_{IH}, at least for standard 2¼ Cr-1Mo steel [2].

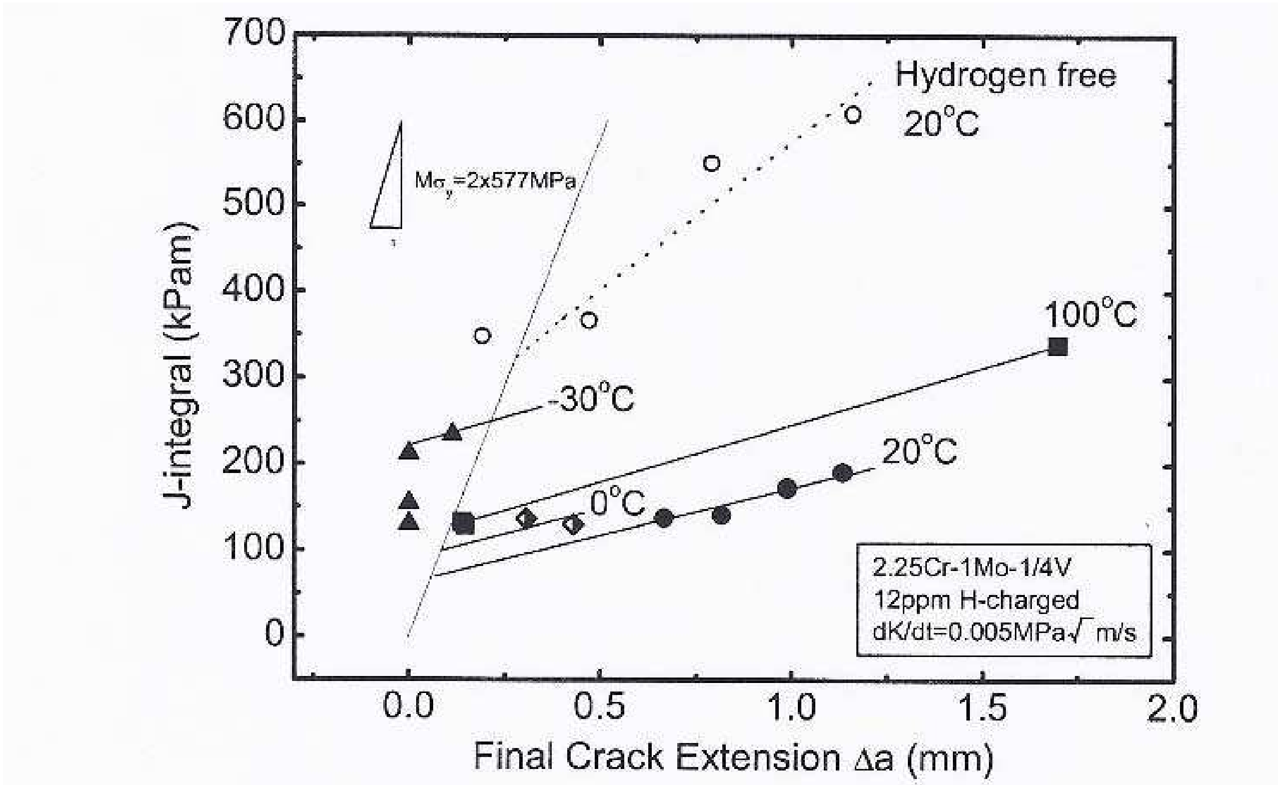


Figure 19 (after Wada et al. [18])

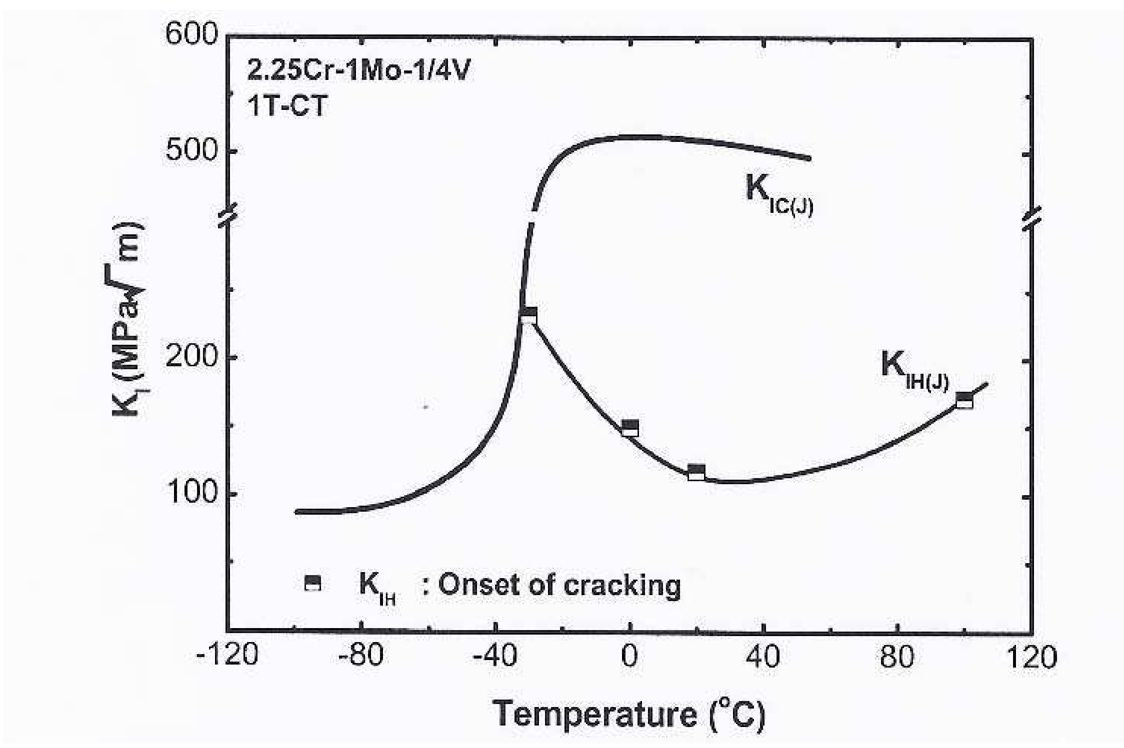


Figure 20 (after Wada, et al. [18])

The results in Figures 19 and 20 show that this V-modified Cr-Mo base plate is susceptible to IHAC at 20 °C, as well as at 100 °C and 0 °C. H cracking is not indicated for the lowest temperature examined (-30 °C), which appears to be in the midst of the H-free ductile-to-brittle transition regime. For the three higher temperatures, IHAC susceptibility is modest, with K_{IH} at or above 110 MPa√m. Such K_{IH} values were determined by the multiple-interrupted specimen method for this V-modified steel (see Figure 19) because comparison between load versus CMOD plots for H_2 charged and H-free specimens did not reveal a definitive difference, due to excessive plastic deformation at these relatively high stress–intensity levels. Notably, the values of K_{IH} shown in Figures 19 and 20 for Cr-Mo-V are four to seven times higher than the typical threshold stress-intensity factors for IHAC in conventional 2¼Cr-1Mo steel (see Figures 1 and 2, and references [2] and [3]).

The upper-shelf $K_{IC(J)}$ values for H-free 2¼Cr-1Mo-0.25 V in Figure 20 are abnormally high and not supported by specific data or experience with this class of steel. The $K_{IC(J)}$ for H-free steel, suggested by a 0.2 mm crack growth offset in the R-curve of Figure 19, is 280 MPa√m (J_{IC} = 0.35 MPa·m). This more reasonable fracture toughness is above the K_{IH} for the H-charged steel at 100 °C, so some IHAC is indicated. These results suggest that this V-modified steel is more resistant to IHAC than conventional Cr-Mo steels; this may be either an intrinsic improvement in K_{IH}, or a false indication due to substantially reduced da/dt from reversible H trapping at V_XC_Y. Only a single loading rate (0.005 MPa√m/s) was examined, similar to the standard value used for extensive experiments with conventional Cr-Mo steels (Figure 13), and not accounting for the reduced diffusivity of H in trap-rich V-modified steel. The early stage of IHAC was not accurately captured by the multiple specimen method, as suggested by the data shown in Figure 19. The exact microscopic mechanism of IHAC was not reported.

In 2006, a data sheet published by Japan Steel Works characterized the IHAC resistance of submerged arc weld metal of 2.59Cr-1.01Mo-0.36V-0.09C (X-bar = 7) [19]. The σ_{YS} was reported to equal 577 MPa, and σ_{UTS} equaled 688 MPa, as developed by heat treatment at 705 °C for 8 h. Compact tension specimens (25.4 mm thick) were fatigue precracked, then H charged for 48 h in 19.8 MPa H_2 at 482 °C to produce an initial bulk H content of 13.5 wppm H. The slow-rising CMOD experiment was employed, using both the load deviation and multiple-specimen J-R curve methods to establish K_{IH}. A single loading rate was used, with dK/dt estimated to equal 17 MPa√m/h; however, the precise loading rate was not specified. The amount of H remaining in the slow-fractured compact tension specimens was 9.0 to 9.1 wppm. The following properties were reported [19]:

— K_{JIC} = 280 MPa√m for H-free weld metal at 23 °C;

— K_{IH} = 88 and 95 MPa√m at 23 °C, from the load deviation method;

— $K_{JIH\text{-}0.2}$ mm offset = 104 MPa√m at 23 °C, from the multiple-specimen J-Δa R-curve method.

Supporting data are given in Figure 21 for K_{IH}, and in Figure 22 for the J-Δa-based offset value of K_{JIH} [18]. The reported values of K_{IH} for this weld metal, 88 and 95 MPa√m, are in good agreement with the value measured for a V-modified base plate (Figure 20). In each case, K_{IH} is less than K_{IC}, suggesting that IHAC occurred, albeit at a relatively high initial-total-dissolved H concentration of 12 to 14 wppm. Nonetheless, the absolute values of K_{IH} are substantially higher than those reported for conventional Cr-Mo steel at about half of this hydrogen concentration (see Figures 1 and 2, and References [2] and [3]). The results presented in Figures 20 through 22 are the result of high-quality experimentation applied to modern heats of V-modified Cr-Mo steel. As such, they are well suited for comparison to the results of the present work being carried out for the API task group.

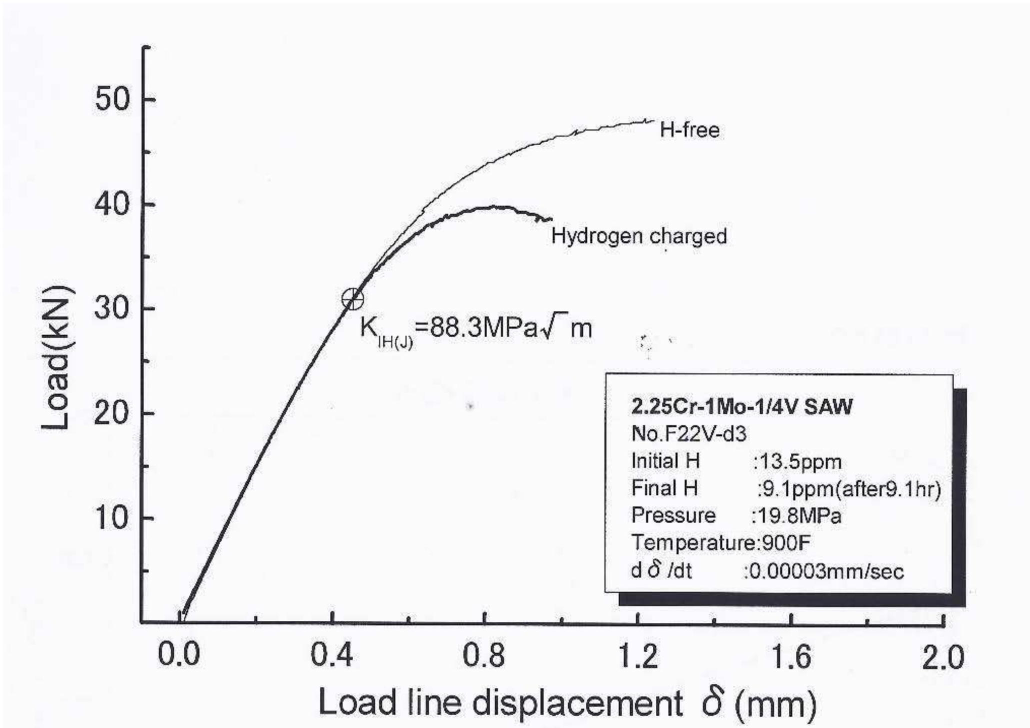


Figure 21 (Japan Steel Works data sheet [19])

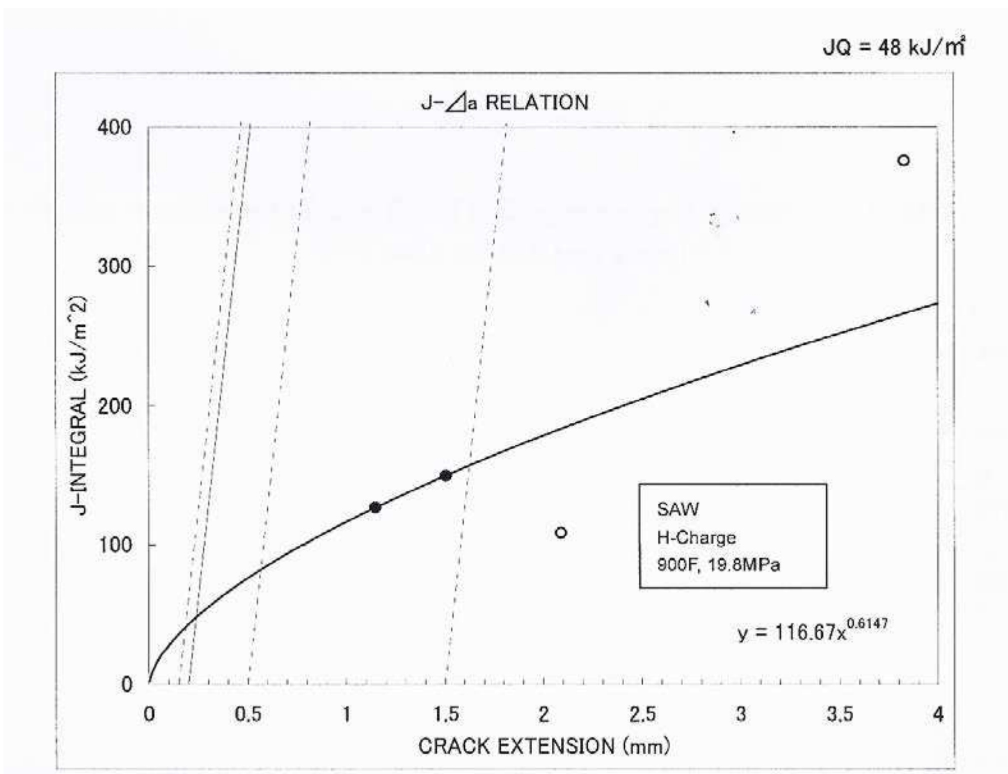


Figure 22 (Japan Steel Works data sheet [19])

A recent series of papers by a large group of Japanese authors, collaborating under the auspices of the Japan Pressure Vessel Research Council's Subcommittee on Hydrogen Embrittlement, provides a modern characterization of the IHAC resistance of V-modified 2¼ Cr-1Mo base plate [20–23]. This work characterized the four steels represented in Figure 23 [23].

Steel	C	Mn	P	S	Si	Cr	Mo	Ni	V	Nb	Sn
A	0.15	0.55	0.009	0.003	0.08	2.40	1.08	0.15	<0.001	0.001	0.006
B	0.15	0.56	0.016	0.002	0.13	2.39	1.05	0.15	<0.001	0.001	0.011
C	0.14	0.56	0.021	0.002	0.24	2.39	1.07	0.14	<0.001	0.001	0.022
V	0.14	0.57	0.010	0.002	0.09	2.41	1.06	0.16	0.32	0.033	0.006
ASTM A387 Grade 22 class 2	0.04–0.15	0.25–0.66	<0.035	<0.035	<0.5	1.88–2.62	0.85–1.15	—	—	—	—
ASTM A387 Grade 22 V	0.09–0.18	0.25–0.66	<0.02	<0.015	<0.13	1.88–2.62	0.85–1.15	<0.28	0.23–0.37	<0.08	—

Steel	J-factor	Heat treatment	YP (MPa)	TS (MPa)	EL, (%)	RA, (%)	FATT (°C)
A	95	PWHT	472	610	26	77	−77
		PWHT + SCHT	464	601	27	78	−58
B	186	PWHT	477	621	24	75	−39
		PWHT + SCHT	476	619	25	74	−24
C	344	PWHT	497	651	24	71	−5
		PWHT + SCHT	488	651	25	70	45
V	106	PWHT	609	716	22	75	−62
		PWHT + SCHT	602	710	26	75	−56
ASTM A387 Gr. 22 Cl. 2			>310	515–690	>18	>45	—
ASTM A832 Gr. 22 V			>415	585–760	>18	>45	—

Note: J-factor = (Si + Mn)(P + Sn) × 10^4, JIS-Type 10 (14ϕ GL = 50 mm), 1/2 t-Transverse.

Figure 23 (after Shimazu et al. [23])

The Cr-Mo-V base plate composition was tempered at 730 °C for 30 minutes and post-weld heat treated at 705 °C for 8 h. Selected specimens were also laboratory step cooled to simulate long-term temper embrittlement. Compact tension specimens, 25.4 mm thick with side-grooves, were exposed to high-pressure H_2 (450°C, 15 MPa, 48 h). This charging condition produced a bulk hydrogen concentration of 5.6 wppm, prior to IHAC testing, and 8.4 wppm measured from a broken compact tension specimen, each for the Cr-Mo-V steel labeled "V" in Figure 23. For the conventional Cr-Mo steel ("A" in Figure 23), this same charging condition produced 2.7 wppm H (initial), and 1.0 wppm H after IHAC testing. Specimens were subjected to slow-rising CMOD loading at an initial dK/dt of 18 MPa√m/h and at 23 °C. Particular emphasis was placed on dcPD measurement of the onset of IHAC at K_{IH}, using a method similar to that employed in the current API task group work in order to eliminate the effect of crack tip plastic strain in rendering a false indication of IHAC growth [21–23]. Overall, this testing protocol contained many of the elements developed during the JIP program in the mid-1990s [2].

Results are tabulated in Figure 24, and J versus stable crack extension R-curves are shown in Figure 25 for conventional (steel A, top) and V-modified (steel V, bottom) 2¼Cr-1Mo base plates [22]. The conventional Cr-Mo steels are prone to substantial IHAC, even at a low total H concentration of 2.7 wppm. In contrast, the V-modified steel is highly resistant, but not immune, to IHAC in the presence of a total H concentration of between 5.6 and 8.4 wppm (for reference in these figures, J values of 20, 250, and 450 kJ/m^2 correspond to K values of 67, 236, and 315 MPa√m). Figure 26 presents a summary of all IHAC data for steels A, B, C, and V [21–23], with K_{IH} plotted as a function of the FATT, which indicates the role of steel purity on the interaction of temper embrittlement and H embrittlement [2,4]. The improved IHAC resistance of the V-modified Cr-Mo steel is readily apparent, compared to a modern high purity heat of conventional

Cr-Mo steel, each exhibiting a low FATT of about -60 °C. The values for K_{IH} are 220–230 MPa√m, well above the threshold of 110 MPa√m reported by Wada for 2¼ Cr-1Mo base plate at 23 °C (Figure 20).

Steels		K_{IC} and K_{IH} (MPa√m)	
		H-Free	H-Charged
A	as-PWHT	273, 230*	68
	PWHT+SCHT	289, 248*	65, 50*
B	as-PWHT	247	66
	PWHT+SCHT	283	67
C	as-PWHT	293	53, 54*
	PWHT+SCHT	288	47, 53*
V	as-PWHT	276	228
	PWHT+SCHT	256, 264*	224

Data with asterisks * denote that experiment was performed under load control.
Fracture toughness was converted from the J-integral calculation based on the basic test method per ASTM E1820-11.

Steels		dJ/da (MN/m^2)	
		H-Free	H-Charged
A (J-factor = 95)	as-PWHT	235	21
	PWHT+SCHT	431	24
B (J-factor = 186)	as-PWHT	409	29
	PWHT+SCHT	324	23
C (J-factor = 344)	as-PWHT	286	33
	PWHT+SCHT	346	12
V (J-factor = 106)	as-PWHT	280	179
	PWHT+SCHT	336	299

Experiment was performed under displacement control.

Figure 24 (after Konosu et al. [22])

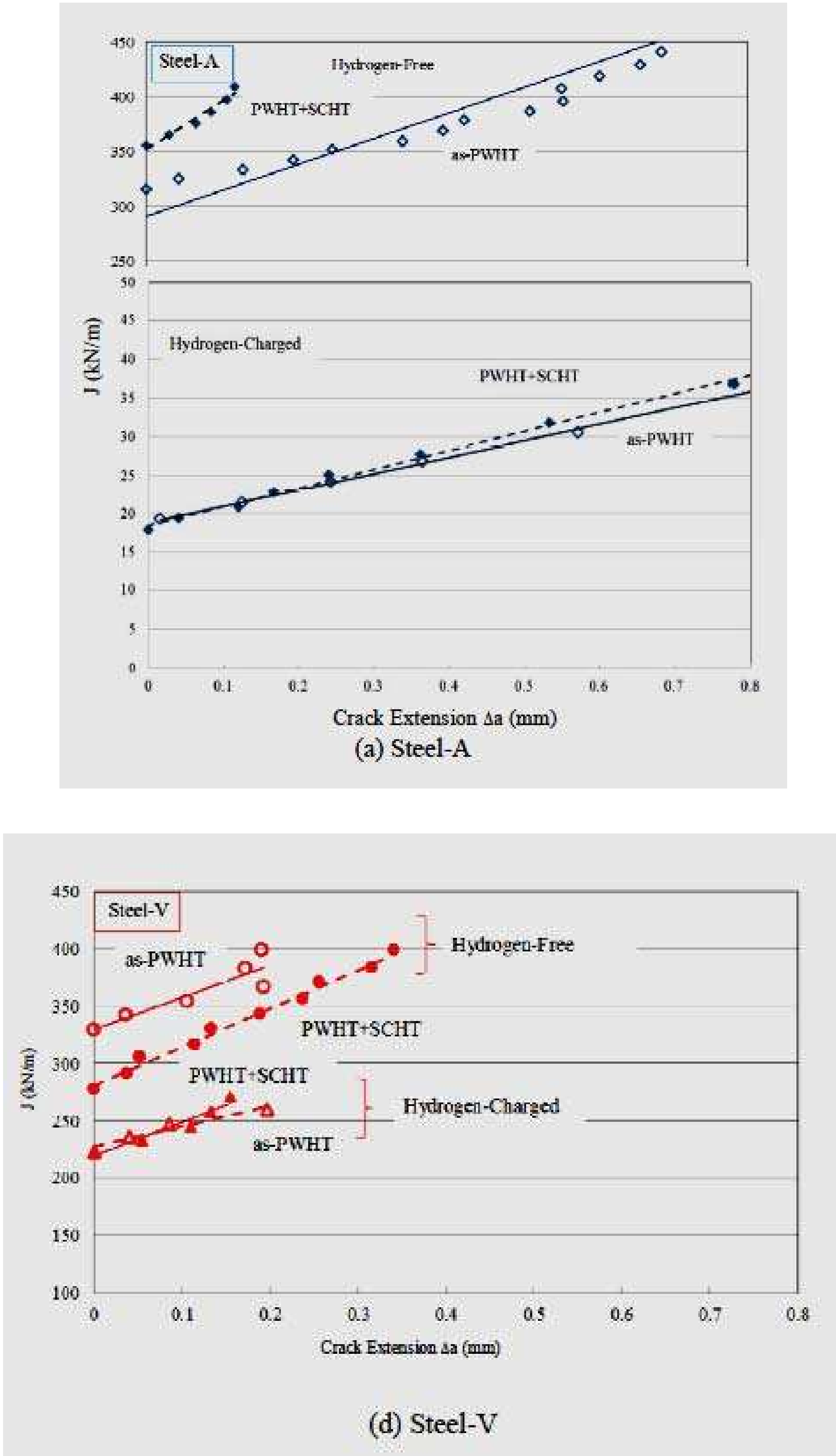


Figure 25 (after Konosu et al. [22])

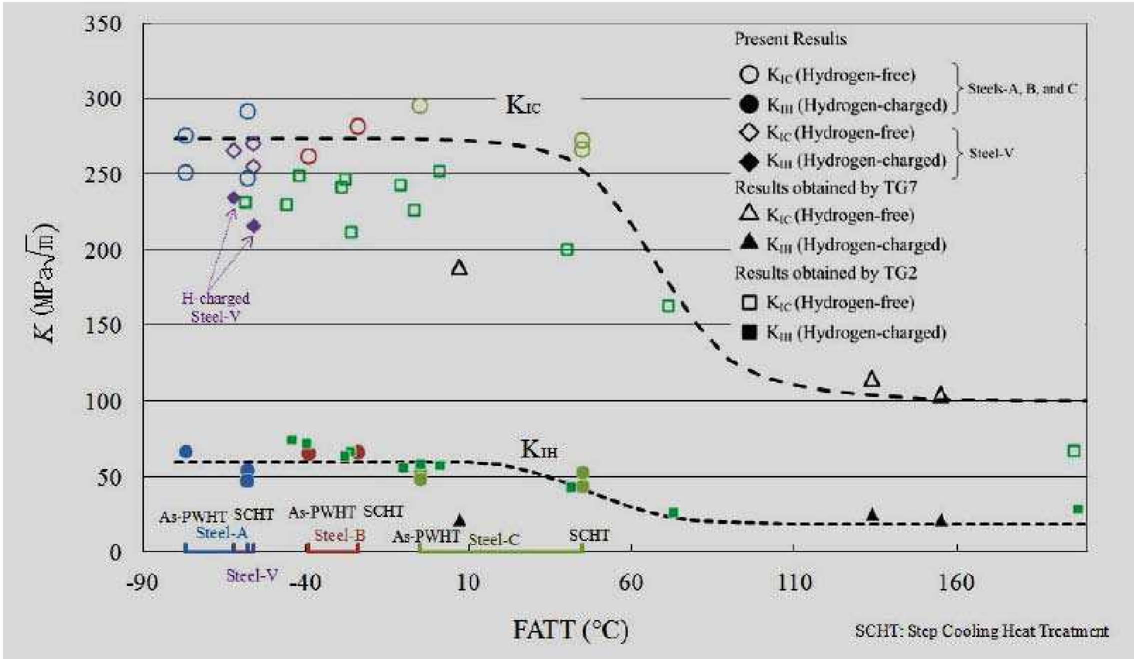


Figure 26 (after Shimazu et al. [21–23])

The microscopic fracture mode for the V-modified steel (V in Figure 21) was always transgranular after H charging. The authors ascribed this to microvoid-based fracture, similar to that observed for H-free 2¼Cr-1Mo-0.25V, but with a reduced dimple depth due to H. Typical fractographs are given in Figure 27 for H-charged V-modified steel [21]. A more detailed fractographic analysis is required to establish the precise mechanism(s) for transgranular IHAC at relatively high stress intensity levels for V-modified steel.

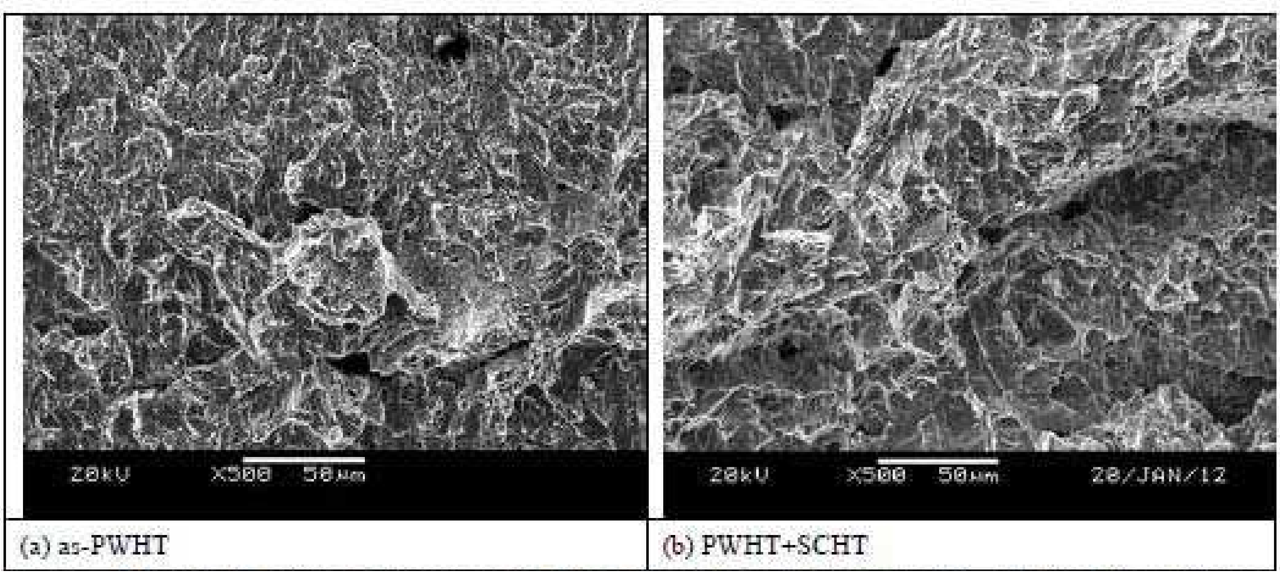


Figure 27 (after Shimazu et al. [21,23])

2.3 Hydrogen Solubility, Diffusivity, and Trapping in V-modified Cr-Mo Steel

A detailed analysis of hydrogen solubility, diffusivity, and trapping in V-modified Cr-Mo steel is centrally important to designing fracture-mechanics experiments, interpreting the resulting IHAC, and implementing estimation of minimum pressurization temperature and other fitness-for-service models [3]. A review of the pertinent literature is beyond the scope of this present analysis.

Pillot and co-workers reported a current tabulation of H solubility and diffusivity in 2¼ Cr-1Mo-0.25V, as shown in Figure 28 [15,24]. For 2¼Cr-1MoV, the diffusion law in the 2012 paper was given as D (m^2/s) = 8.00×10^{-8} exp (-24,975/RT) [24], with the activation energy as J/mol. The difference compared to the result in Figure 28 likely stems from the challenging nature of H diffusion characterization in a trap-rich microstructure. The solubility relationship shown in Figure 28 is identical to that reported previously [24].

Grade	Diffusion law D(T) ($m^2.s^{-1}$)	Solubility law S(T) ($mol.m^{-2}.Pa^{-0.5}$)	Reference
2¼Cr1Mo	$2.28*10^{-7}$.exp(-16936/RT)	0.592.exp(-27079/RT)	27
2¼Cr1MoV	$6.70*10^{-7}$.exp(-30034/RT)	0.036.exp(-6943/RT)	19, 28
3Cr1MoV	$2.50*10^{-7}$.exp(-25346/RT)	$2.59*10^{-2}$.exp(-6182/RT)	27
18-10 type SS	$7.69*10^{-7}$.exp(-53300/RT)	0.151.exp(-4500/RT)	27

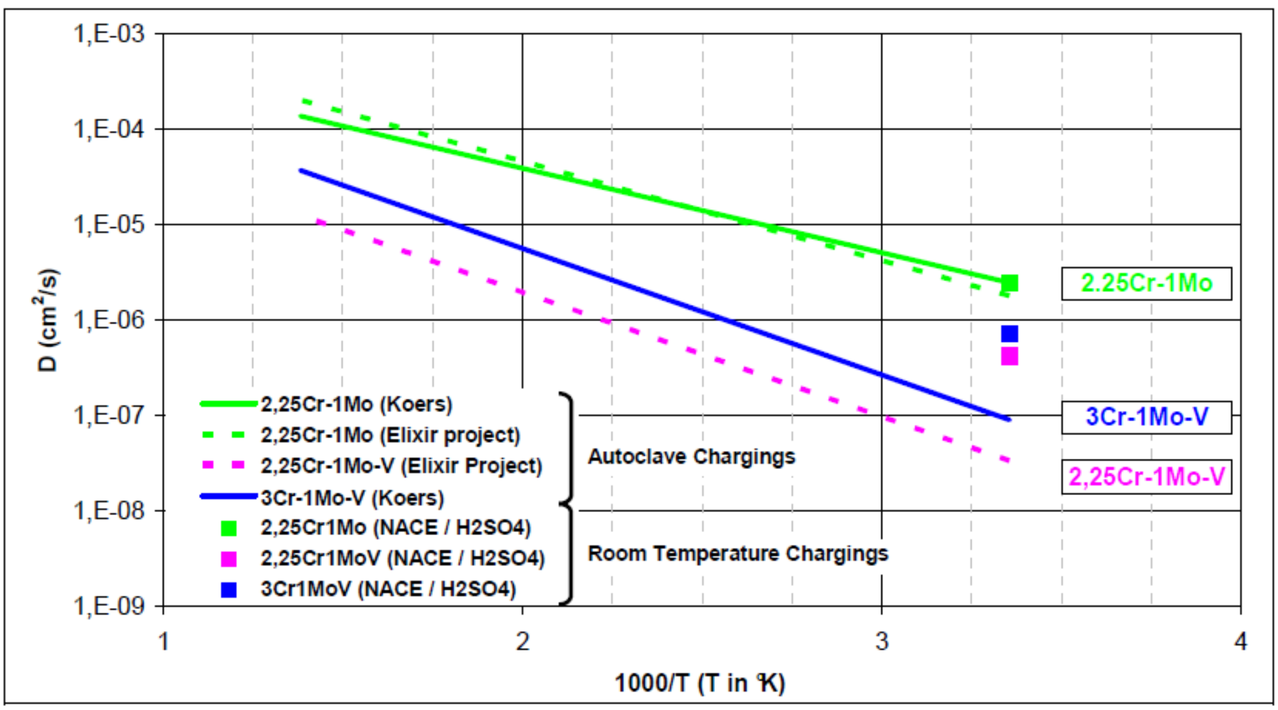


Figure 28 (from Pillot et al. [15])

A detailed thermal desorption spectroscopy analysis, including several heating rates, is necessary to establish precise H-trap binding energies and site coverages for a given steel microstructure [25,26]. Several studies have been reported in this regard for conventional and V-modified Cr-Mo steels; however, none are sufficiently detailed or definitive to yield precise H-trap binding energies, or necessary to enable interpretation of the temperature-dependent IHAC in V-modified Cr-Mo [27–33]. An example TDS spectra for a single heating rate is presented in Figure 29 [22]. Specimens sectioned from broken compact-tension specimens, stressed in air at 23 °C for the indicated times, were heated in the thermal desorption spectrometer at 100 °C h^{-1}. The V-modified steel exhibits a larger concentration of dissolved H (area under the curve), and a H desorption peak that occurs at a substantially higher temperature compared to the conventional Cr-Mo steel (about 260 °C for steel V versus 160 °C for steel A). This higher temperature peak suggests relatively strong H trapping, likely associated with V_XC_Y precipitates, in the V-bearing steel, compared to weaker trapping in the conventional Cr-Mo composition. Substantially more detailed TDS work is required to characterize and understand H trapping versus both charging temperature and diffusion temperature for V-modified Cr-Mo steels.

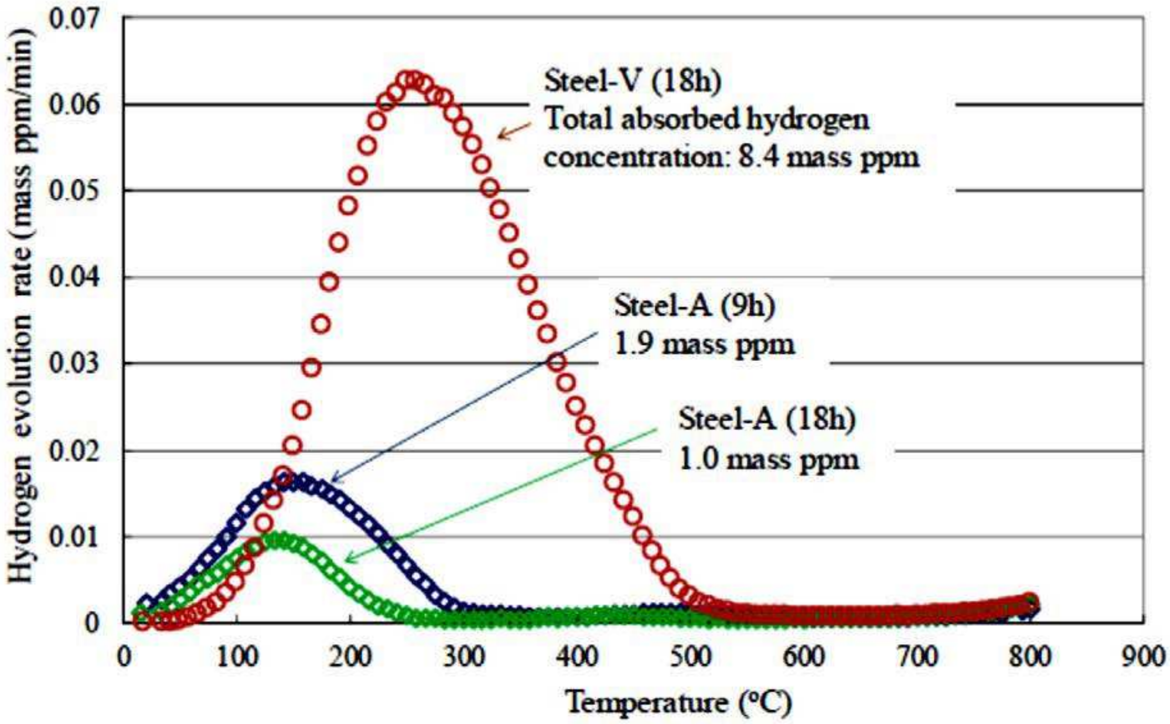


Figure 29 (after Konosu et al. [22])

2.4 Hydrogen Embrittlement of Cr-Mo and Cr-Mo-V Steels in H_2

All IHAC results presented in this review are based on cracking in H precharged specimens during slow-rising displacement loading in moist laboratory air. Discussion during the last two API task group meetings included the possibility of a deleterious effect of loading a H precharged specimen in gaseous hydrogen at 23 °C. Limited experimental results have been reported in the published literature and open-committee reports to enable consideration of the interaction of IHAC and hydrogen environment–assisted cracking (HEAC) in H_2. In general, there has been no systematic published study of the interaction of IHAC and HEAC for a C-Mn or low-alloy steel [2,34]

Watanabe and co-workers presented an early and important result that quantified the extent of the interaction between IHAC and HEAC for a conventional Cr-Mo steel base plate [10]. This 2¼Cr-1Mo steel was a commercial forging of a modern low-J-factor composition (Fe-2.4Cr-1.08Mo-0.47Mn-0.15C-0.005Sn-0.003As-0.006S-0.007P; J = 60) tempered (time and temperature not reported) to an ultimate tensile strength (σ_{uts}) of 758 MPa. Compact tension specimens were H precharged in 9.8 MPa H_2 at 482 °C for 48 h. Measured H concentrations were 4.4 to 4.8 wppm for this steel strength and charging temperature-pressure. Separate outgassing experiments demonstrated that this level of H was retained in coupons exposed in moist air at 23 °C for loading times up to 5 h, which was the reported time of a standard IHAC experiment. K_{IH} values are presented in Figure 30 for CT specimens stressed in moist air, as well as in high-pressure H_2 at either 10.3 or 20.7 MPa and at 23 °C for each case [10]. Figure 30 establishes the important effect of applied loading rate given as a crosshead (grip) displacement rate. In a separate slide in the Reference 8 presentation, the authors state that a crosshead speed of 0.0025 mm/min is equivalent to an initial dK/dt of about 6.6 MPa√m/h, which includes the compliance characteristics of the specific test machine and grip system. Several experiments in Figure 30 were conducted at the typical crosshead speed of 0.005 to 0.006 mm/min, which, according to this calibration, is equivalent to an initial dK/dt of 13–16 MPa√m/h.

The results in Figure 30 demonstrate substantial hydrogen embrittlement, but do not prove a deleterious interaction between IHAC and HEAC for this 2¼Cr-1Mo steel. For all cases considered, measured K_{IH} was substantially less than K_{IC} (which is on the order of 300 MPa√m at 23 °C, but not specifically stated for this modern forging). Hydrogen cracking was indicated to be consistently intergranular. First, consider IHAC based on K_{IH} for H precharged specimens loaded in moist air. At the typical crosshead displacement rate of 0.005 mm/min (dK/dt = 13 MPa√m/h), the K_{IH} is 45 to 56 MPa√m (a separate plot in Reference 8 reported K_{IH} values of 26 and 28 MPa√m for presumably this crosshead speed; these values are lower than those represented in Figure 30). For loading in moist air, increasing crosshead speed to 130 MPa√m/h or 1,300 MPa√m/h resulted in K_{IH} increasing to 95 MPa√m and 120 MPa√m, respectively, and as expected for IHAC in conventional Cr-Mo steel [2]. The K_{IH} values measured for the loading of precharged CT specimens in the two high pressures of H_2 are identical to the value measured for IHAC in moist air, when the comparison is made at this relatively slow loading rate of 0.005 mm/min (13 MPa√m/h). As such, there is no evidence supporting a deleterious interaction between IHAC and HEAC at this loading rate.

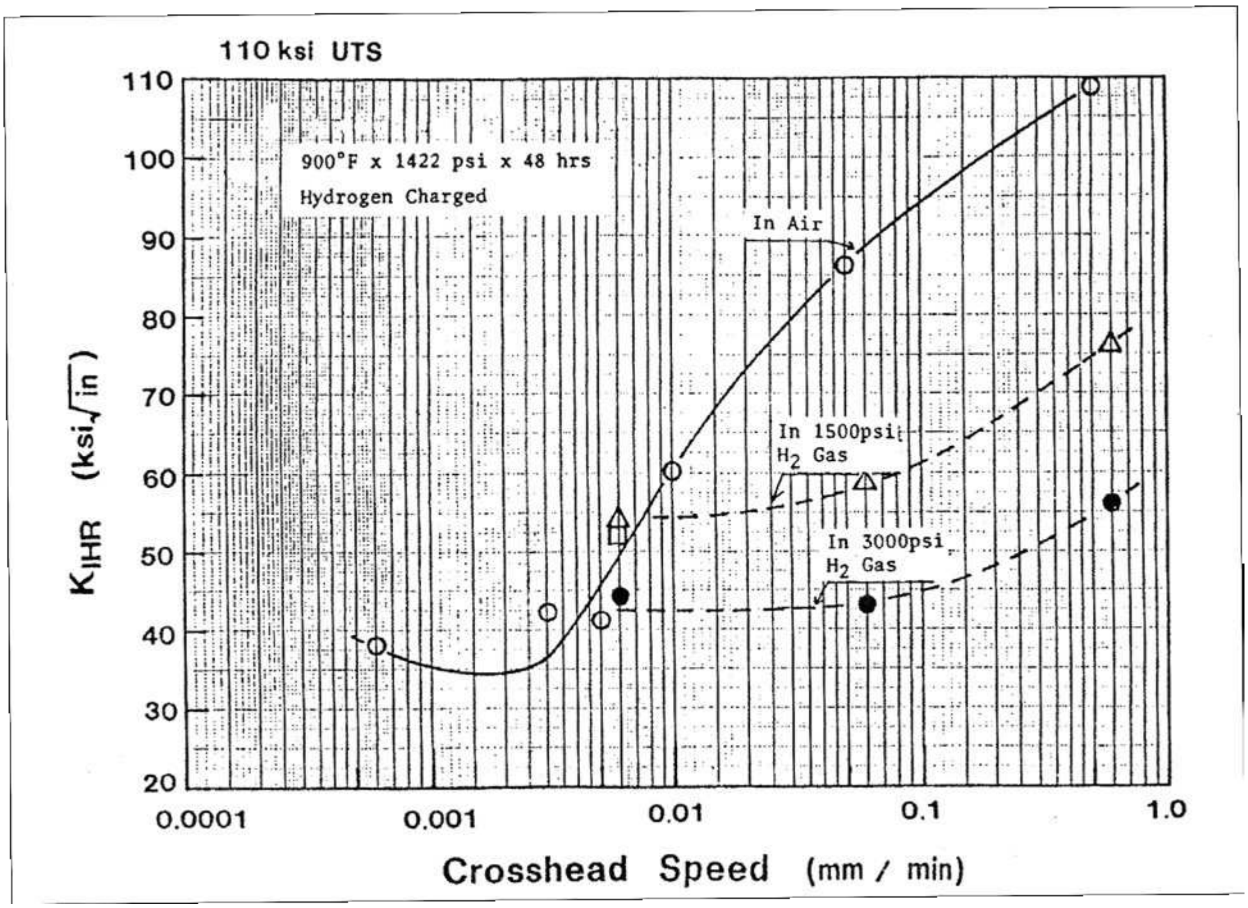


Figure 30 (after Watanabe et al. [10])

Figure 30 demonstrates that, as the loading rate increases to relatively high levels, K_{IH} values measured with H precharged specimens are lower for cracking in each pressure of H_2 compared to IHAC in moist air. The deleterious effect of loading in H_2 increases as H_2 pressure increases, which is expected for HEAC only [34]. The results of these experiments at higher loading rates are not sufficient to establish a deleterious interaction of IHAC and HEAC because literature data show that similar steels are susceptible to hydrogen embrittlement under slow-rising displacement loading when stressed in high-pressure H_2 without H precharging. Control experiments, with uncharged conventional Cr-Mo steel stressed at various loading rates in high-pressure H_2, were not reported in Reference 8 and are not otherwise available in the literature.

Specific data that demonstrate significant HEAC susceptibility are presented in Figure 31 for two low-strength C-Mn steels [34–36], and in Figure 32 for two moderate-strength alloy steels [34,37] (the slow-rising displacement rates used for the experiments in Figure 31 are noted, but the precise meaning of this rate, and the equivalence to dK/dt, are not known). If the expected approximate relationship between crosshead speed and dK/dt holds [2], the two displacement rates in Figure 31 correspond to relatively rapid dK/dt levels of 95 and 2450 MPa√m/h. The results in Figure 32 were produced by static displacement (decreasing stress intensity) testing to establish a crack arrest threshold. The rising K-Δa R-curves that yielded the K_{IH} values for A516 steel are shown in Figure 33 [39]. The more deleterious effect of slow-rising displacement, compared to static displacement crack arrest loading, on the threshold for IHAC and HEAC is discussed elsewhere [2,38].

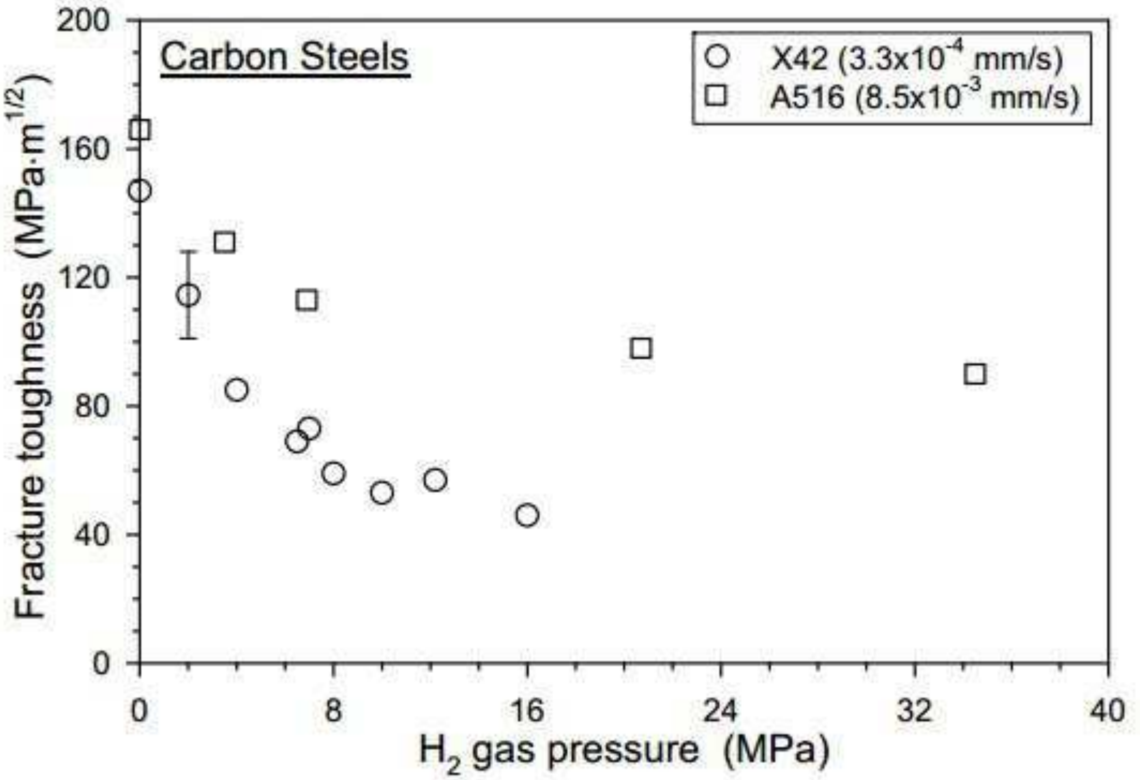


Figure 31 (after San Marchi and Somerday [34])

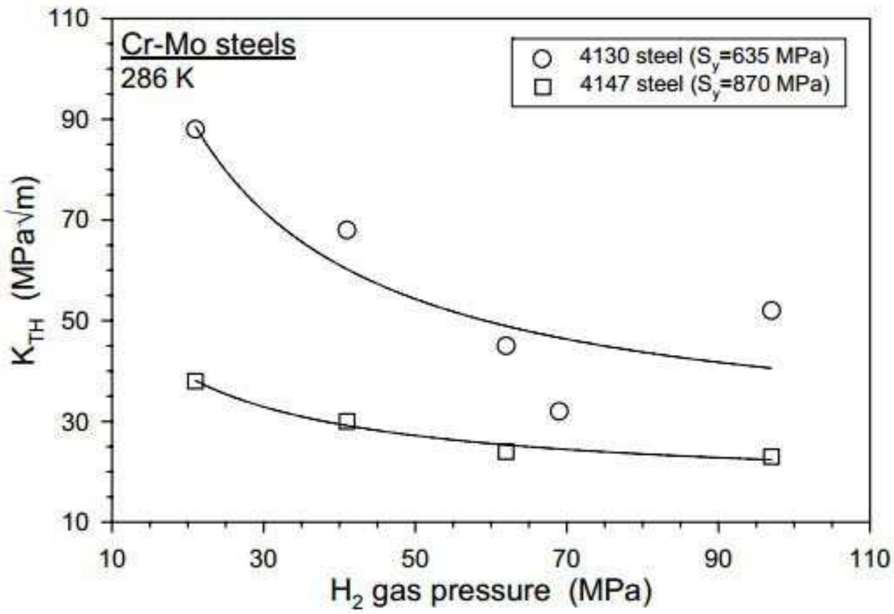


Figure 32 (after San Marchi and Somerday [34])

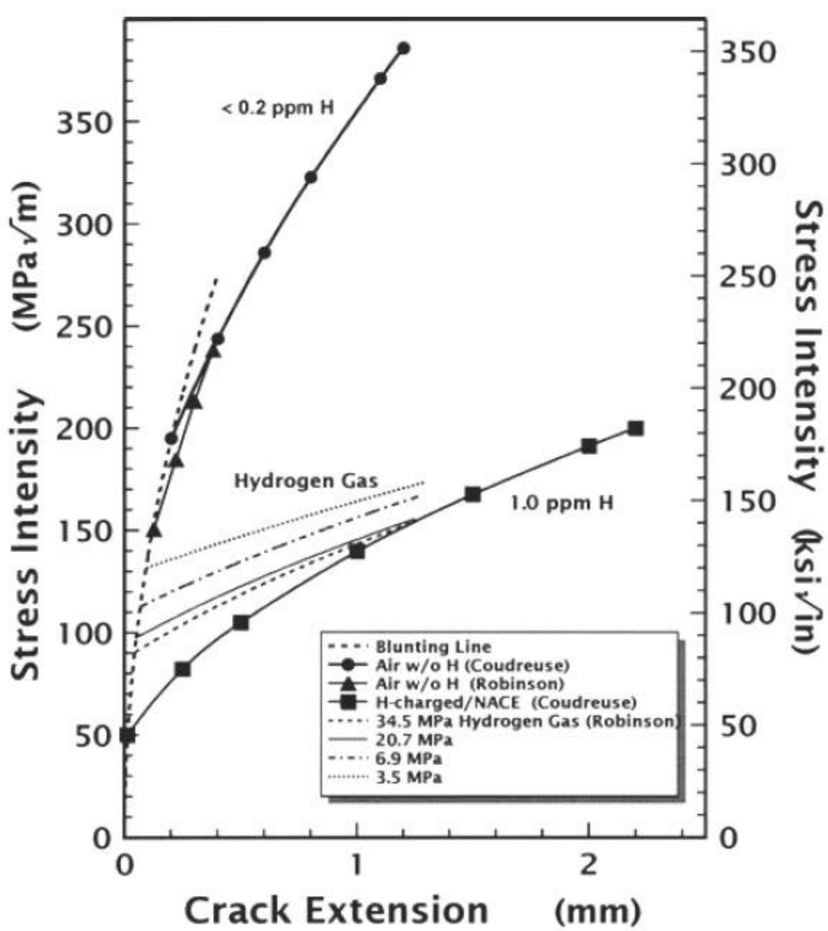


Figure 33 (after Gangloff [39])

Figure 34 compares the HEAC resistance of the steels shown in Figures 31 through 33 with the limited experimental data obtained for conventional and V-modified 2¼Cr-1Mo steels. Watanabe's data from Figure 30, taken for the lowest loading rate applied to H precharged Cr-Mo steel stressed in H_2, are shown by **+**, and the K_{IH} for this H precharged steel stressed at the same dK/dt in moist air is shown by ◘ [8]. A single result for A516 steel, precharged in an electrochemical environment to produce 1.0 wppm H, then subjected to slow-rising CMOD in moist air, is shown by ●, taken from the appropriate value tabulated in Figure 11 [12]. The data for low-strength X42 from Figure 31 are represented by ----- [36]. Finally, K_{IH} values were recently measured for 2¼Cr-1Mo-0.25V base plate subjected to slow-rising CMOD loading in high-pressure H, without H precharging; results are represented by (◆) for H_2 pressures of 0.34 and 103 MPa [40]. The initial dK/dt was 24 MPa√m/h.

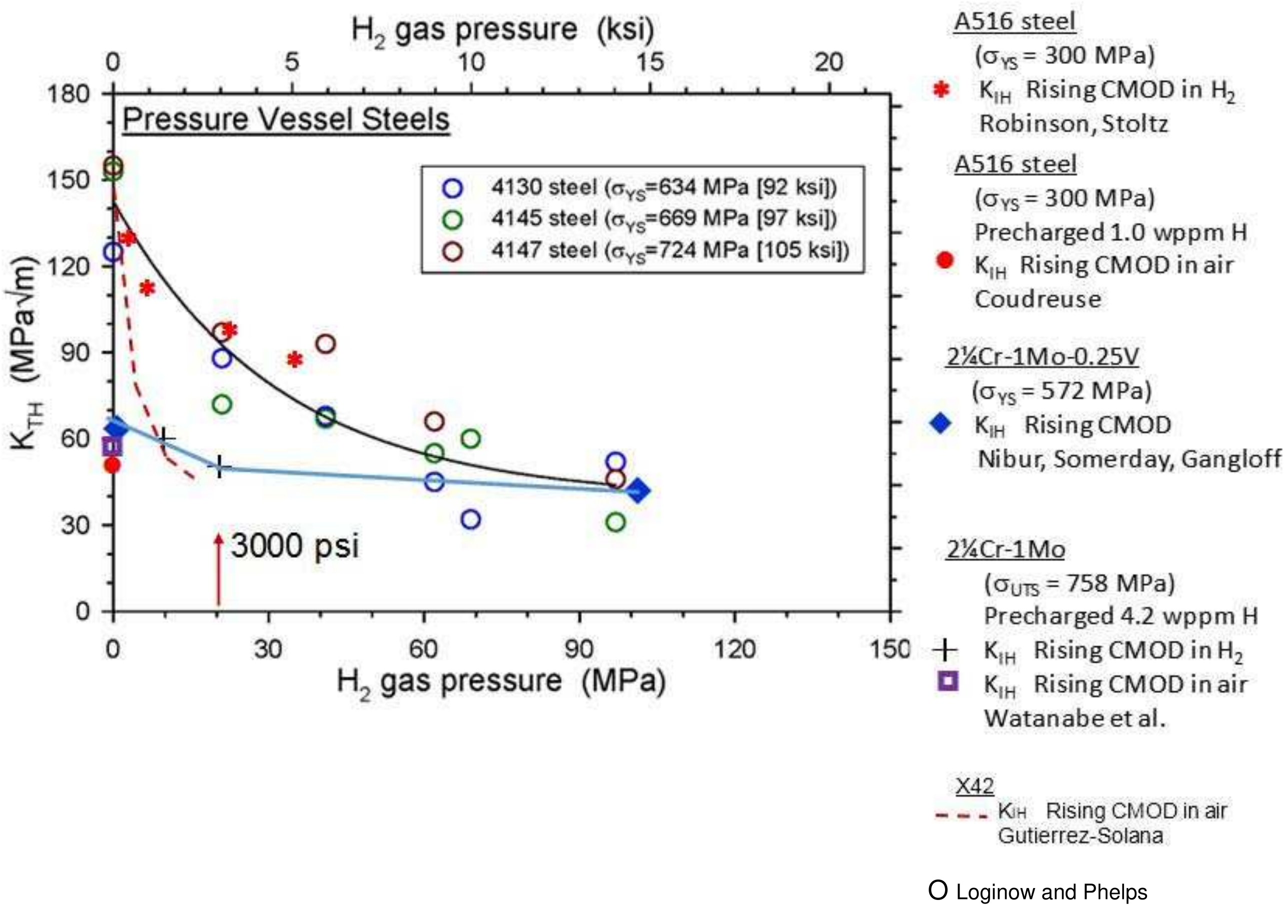


Figure 34 (after Nibur, Somerday, and Gangloff [40])

The results in Figure 34 do not support a novel deleterious interaction of IHAC and HEAC for Cr-Mo and Cr-Mo-V steels. Either H precharging, with stressing in moist air, or stressing in high-pressure H_2, without H precharging, is each capable of producing substantial H embrittlement in conventional Cr-Mo steel. There are no data available that show a uniquely low K_{IH} for IHAC plus HEAC when combined. This statement is true, but tempered by the fact that there are no K_{IH} results in Figures 30 or 34 that are specific to uncharged conventional Cr-Mo stressed in high-pressure H_2. The behavior of similar steels, particularly low-strength X42 steel (---- in Figure 34), suggests that Cr-Mo steels will exhibit low K_{IH} in high-pressure H_2 when stressed at the dK/dt typical of IHAC experiments. The most likely interpretation of the data in Figure 30, which show more severe IHAC + HEAC for conventional Cr-Mo, is that this behavior is dominated by HEAC alone and not by the IHAC + HEAC interaction. The crack tip H diffusion distance for HEAC (~1 to 5 μm) is substantially less than that required for IHAC. As such, the equilibrium threshold for K_{IH} for HEAC is sustained to higher dK/dt, compared to IHAC, before H cracking becomes kinetically limited. This significant HEAC is likely reduced as the H_2 pressure falls to levels typical of reactor shutdown conditions. Simply stated, the lower K_{IH} values for H-charged Cr-Mo steel in Figure 30 are due solely to the embrittling effect of

H_2, which was sustainable to higher loading rates. Control data do not exist for conventional uncharged Cr-Mo specimens, stressed in high-pressure H_2 at these faster loading rates; as such, this interpretation is speculative.

Finally, K_{IH} values for uncharged 2¼Cr-1Mo-0.25V base plate stressed in high-pressure H_2 (◆ in Figure 34) are substantially less than the thresholds typical of IHAC from precharged H, as summarized in previous sections. Significant HEAC is observed for a very low H_2 pressure (0.34 MPa), which could be relevant to reactor shutdown and startup service. This HEAC susceptibility may be akin to the significant IHAC in V-modified steels, produced by room-temperature H charging (see Figures 15 and 16), with each behavior perhaps traced to H interaction with V_XC_Y trap sites. This result justifies additional experiments to determine the H_2 pressure dependence of K_{IH} for V-modified Cr-Mo. Such work would include confirmation that there is no deleterious interaction between IHAC and HEAC, and quantification of slow crack growth rates that may be impacted by residual-reversible H trapping at V_XC_Y.

2.5 Conclusions

Review of the existing literature regarding the internal hydrogen-assisted cracking (IHAC) resistance of V-modified 2¼Cr-1Mo type steel establishes the following conclusions.

— Individual experiments establish that V-modified Cr-Mo base plate and weld metal are highly resistant to IHAC at 23 °C, compared with conventional 2¼Cr-1Mo, with K_{IH} equal to a substantial fraction of K_{IC} and a resolvable but modest lowering of the J versus Δa R-curve. For this comparison, the conventional and V-modified steels were low impurity-low FATT, and IHAC improvement is associated with H trapping at V_XC_Y precipitates.

— Literature results published to date do not provide any evidence that predissolved H lowers the resistance of Cr-Mo-V steel to unstable-fast fracture, consistent with the very low H-free FATT values exhibited by such modern-pure steels.

— The most rigorous fracture-mechanics experiments, conducted after about 2002 and with commercial heats of 2¼Cr-1Mo-0.25V base plate and weld metal, yield the following values of K_{IH} for loading at a relatively rapid rate (dK/dt) of 18 MPa√m/h predominantly at 23 °C. These experiments accurately computed an elastic-plastic stress intensity versus crack extension to yield both K_{IH} and a sense of the K_J versus stable crack extension "R-curve."

Modern Fracture Mechanics Characterization of the Rising-CMOD Threshold Stress Intensity for IHAC in Conventional and V-modified Cr-Mo Steel

Stressing Temperature °C	$C_{H\text{-}Total}$ Initial--final wppm	Product Final Heat Treatment	σ_{UTS} MPa	FATT °C	dK/dt MPa√m/h	K_{IH} MPa√m	Source	Published
2¼Cr-1Mo								
23 °C	2.0--?	BP WM	582 to 649	-28 to -90	25	50	JIP Gangloff	2011 [3]
23 °C	6.0--?	BP WM	582 to 649	-28 to -90	25	22	JIP Gangloff	2011 [3]
2¼Cr-1Mo-0.25V								
23 °C	12--9	BP 705 °C 8 h	663	-78	18	115	Wada et al.	2002 [18]
100 °C	12--?	BP 705 °C 8 h	663	-78	18	170	Wada et al.	2002 [18]
23 °C	13.5--9	WM 705 °C 8 h	688	-28 (40 ft-lb)	18	88	Wada et al.	2006 [19]
23 °C	5.6--8.4	BP	716	-62	18	224 to 228	Shimazu et al.	2013 [21–23]

BP = Base Plate WM = Weld Metal

— No single study of IHAC in 2¼Cr-1Mo-0.25V base plate or weld metal is sufficiently comprehensive and quantitative to support fracture-mechanics modeling of pressure vessel fitness for service, or a minimum pressurization temperature, centered on the potentially deleterious effect of elevated-temperature high-pressure H_2 charging of H.

— The following issues limit the existing IHAC database for V-modified Cr-Mo, particularly the 2¼Cr-1Mo-0.25V composition:

  — It is challenging to resolve an accurate K_{IH} for the onset of IHAC propagation in the presence of significant crack tip plastic deformation typical of an IHAC-resistant, moderate-strength steel. Characterization of slow crack growth rates typical of strong-reversible H-trap interaction is equally difficult.

  — The effect of a slow loading rate on K_{IH} for IHAC in a trap-laden microstructure, which reduces the mobility of diffusible H, has not been established experimentally.

  — The effect of very high H concentration on IHAC, dissolved in the steel at elevated temperature and trapped at various microstructural sites during cooling, has not been established.

  — The effect of stressing temperature on IHAC has not been sufficiently determined; particularly when coupled with slow loading rate, precision measurement of K_{IH}, and verification of the amount of H retained during loading. Temperatures in the range from -25 °C to 200 °C are of interest to MPT and IHAC mechanism determination.

  — The effect of weld metal microstructure, compared to base plate and as a function of temperature, has not been established; particularly coupled with slow loading rate, precision measurement of K_{IH}, and verification of the amount of H retained during loading.

— The mechanism by which H trapping at V_XC_Y reduces susceptibility to IHAC in V-modified Cr-Mo is not understood, in part due to uncertain H-trap binding energies to such carbides compared with values for H localization at bainite interfaces and within the crack tip stress field.

— Limited data suggest that susceptibility to IHAC is promoted in V-modified Cr-Mo steel when H charging is performed at ambient temperature and using a high-H fugacity environment that could saturate moderate to strong binding energy H traps. This behavior must be better established.

— Initial data show that uncharged 2¼Cr-1Mo-0.25V base plate is susceptible to hydrogen embrittlement under slow-rising displacement loading in high-pressure H_2, conforming with the behavior of low- to moderate-strength steels without vanadium carbide traps. K_{IH} for HEAC in H_2 is substantially less than values typical of IHAC from precharged H, and could be relevant to reactor shutdown and startup service.

— A novel deleterious interaction of IHAC and HEAC is not supported by published data for Cr-Mo and Cr-Mo-V steels. H precharging, with stressing in moist air, or stressing in high-pressure H_2, without H precharging, are both capable of producing substantial H embrittlement in conventional Cr-Mo steel. There are no data available that show a uniquely low K_{IH} for IHAC plus HEAC when combined; low K_{IH} at atypically fast loading rates is most likely due to HEAC alone.

— The ongoing laboratory study, commissioned by API Task Group 934-F on Heavy Wall Pressure Vessel MPT, is addressing IHAC uncertainties for both weld metal and base plate of the 2¼Cr-1Mo-0.25V composition.

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