# Technical Reference on Hydrogen Compatibility of Materials

Austenitic Stainless Steels: A-286 (code 2301)

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## 1. General

A-286 is an iron-base superalloy commonly used for its combination of high-strength and good corrosion resistance at intermediate temperatures. The high-nickel content of A-286 and its sister alloys make them resistant to strain-induced phase transformations. Although also referred to as stainless steel, A-286 is considerably different from the 300-series alloys in that it is strengthened by precipitation of the  $\gamma$ ' phase, Ni<sub>3</sub>(Al,Ti) [1]. Although A-286 can be welded (material specifications exist for welding grades of A-286, e.g. [2]), a modified version of the alloy, called JBK-75, was developed to improve its weldability as well as improve hydrogen compatibility [3]. NASA has developed an alloy called NASA-HR-1, which is based on JBK-75, to improve strength as well as resistance to hydrogen embrittlement, oxidation and corrosion [4]. NASA-HR-1 should not be confused with the Chinese alloy HR-1, which is similar to type 316 stainless steel [5].

The high-nickel and chromium content of the A-286 family of alloys implies high stacking fault energy [6], a characteristic associated with uniform plastic deformation and consequently resistance to hydrogen embrittlement in austenitic stainless steels [7, 8]. The coherent interface of the  $\gamma$ ' precipitates in A-286 and JBK-75, on the hand, tends to enable non-uniform plastic deformation, a feature in austenitic steels that is often used to explain comparatively poor resistance to hydrogen embrittlement [6, 9]. The uniformity of plastic deformation in precipitation strengthened austenitic alloys, however, may be less important in governing hydrogen embrittlement compared to other metallurgical features, such as internal interfaces and second phases that can interact with internal hydrogen.

Two general observations distinguish hydrogen-assisted fracture in the A-286 family of alloys from the single-phase austenitic stainless alloys: (i) A-286 that has been tested in tension in external hydrogen gas is not embrittled [6, 10-14], while A-286 with internal hydrogen (by thermal precharging in hydrogen gas) features a significant reduction in tensile ductility [3, 6, 9, 15], and (ii) JBK-75 (internal hydrogen) and A-286 (external hydrogen) that have been tested at elevated strain rates in tension do not show an increase in ductility compared to low strain rates [14, 16]. These observations could be explained by the tenacious oxide that forms upon aging A-286 (even when aged in reducing environment), which acts as a permeation barrier during the relatively short exposure of tensile tests, if these tensile specimens were machined prior to aging. The presence of an oxide, however, cannot explain the strain rate effect. Holbrook and West on thermally precharged JBK-75, which showed no strain rate effect. Holbrook and West suggested that interactions between hydrogen and dislocations may be different in JBK-75 compared to single-phase austenitic alloys [16].

The mechanisms that contribute to hydrogen embrittlement in the A-286 family of superalloys have not been firmly established. It has been speculated that loss of matrix- $\gamma'$  precipitate coherency during deformation allows hydrogen to accumulate at these incoherent interfaces leading to hydrogen-assisted fracture [6]. Observations of smaller dimple size in the presence of internal hydrogen [6] support the view that hydrogen assists nucleation of microvoids, perhaps at newly incoherent interfaces. Ductile microvoid coalescence, however, competes with intergranular fracture in these alloys in the presence of hydrogen. Intergranular fracture is more prevalent and features less evidence of ductile fracture processes in materials aged for longer times as the number and size of grain-boundary precipitates increases with time [3, 17].

Intergranular fracture is generally attributed to the presence of  $\eta$ -phase (Ni<sub>3</sub>Ti), which precipitates primarily on or near grain boundaries during aging. Heat treatments (and compositional gradients as in welds), for example, that promote precipitation of the  $\eta$ -phase result in higher crack growth rates and lower threshold stress intensity factors in sustainedloading fracture specimens that have been tested in high-pressure gaseous hydrogen [18, 19]. Ductility losses, as determined from tensile tests, however, do not show a dependence on the volume fraction of the  $\eta$ -phase, nor on the degree of intergranular fracture [3, 17]. It has been surmised that failure of tensile specimens in the presence of hydrogen is dominated by crack nucleation in these alloys, and once a crack forms it propagates rapidly along susceptible features such as grain boundaries [17]. For example, precracked tensile specimens of JBK-75 tested in hydrogen gas failed entirely by intergranular fracture, while smooth tensile specimens of the same material in the same conditions failed by microvoid coalescence [13].

While the nature of interactions between hydrogen, dislocations, and the various precipitates in the A-286 family of alloys are not unequivocally known, the data suggest that shorter aging times and lower aging temperatures result in microstructures that are less susceptible to hydrogen effects [3, 17-20]. Fusion weld microstructures may be particularly susceptible to hydrogen because titanium and nickel segregation in the weld may facilitate precipitation of the  $\eta$ -phase [18, 21].

# **1.1 Composition**

Table 1.1.1 lists the compositions of several heats of A-286 used to study hydrogen effects. Welding grades of A-286 specify low silicon and manganese, e.g. [2]. A modified version of A-286, called JBK-75, was developed to improve weldability and hydrogen compatibility [3]; the compositions of several heats of JBK-75 are listed in Table 1.1.2. More recently, JBK-75 has been modified by researchers at NASA to improve strength as well as resistance to hydrogen embrittlement, oxidation and corrosion; this alloy, NASA-HR-1, has additions of tungsten and cobalt in addition to increased nickel and molybdenum content [4].

# 1.2 Other designations

AISI Type 660, UNS S66286 related alloys: JBK-75 (UNS S66285), V-57, NASA-HR-1

# 2. Permeability and Solubility

The permeation and solubility of hydrogen in JBK-75 was found to be independent of heat treatment for conventional solution heat treating and aging cycles [22]. Permeability and solubility generally follow an Arrhenius-type relationship with temperature; Table 2.1 provides these relationships for JBK-75 as well as relationships averaged for several austenitic alloys. Plotting these relationships shows that the superalloys have nominally the same permeability and solubility as the single-phase austenitic stainless alloys, Figure 2.1 and Figure 2.2.

Hydrogen concentration measurements by hot extraction techniques show somewhat different trends from permeation studies. The hydrogen concentration was found to strongly depend on processing conditions for modified A-286 (presumably JBK-75) with internal hydrogen (thermally precharged in hydrogen gas), Table!2.2 [23]. Microstructural details that might

account for the measured difference were not reported or discussed in that study. In another study [15], hydrogen concentration measurements in JBK-75 by hot extraction were reported to be 20% higher than concentrations calculated based on data for austenitic alloys. The source of these discrepancies is not clear, but may be related to additional hydrogen trapped at specific microstructural features, such as precipitate interfaces. Trapping of hydrogen is generally considered to be low in single-phase austenitic alloys, however, further study is necessary to determine if hydrogen trapping is significant in precipitation-hardened stainless steels such as the A-286 family of alloys. While hot extraction techniques determine the total hydrogen in the material, i.e., both trapped hydrogen and mobile hydrogen, the solubility and permeability only depend on lattice or mobile hydrogen, which should not be strongly affected by precipitation in A-286 based alloys [22]. Therefore, the relationships provided in Ref. [24] (when corrected to hydrogen), Table 2.1, should be considered the best conservative (high value) estimate for permeability and solubility when extrapolated to room temperature. Based on available data, an upper bound to the equilibrium concentration of hydrogen in the A-286 family of alloys can be approximated from the recommended solubility relationship.

# 3. Mechanical Properties: Effects of Gaseous Hydrogen

## 3.1 Tensile properties

#### 3.1.1 Smooth tensile properties

Room temperature tensile testing of A-286 and JBK-75 show little or no loss in ductility during straining in hydrogen gas at pressures up to 172!MPa. Tensile specimens with internal hydrogen (by thermal precharging in hydrogen gas), however, show a significant loss in ductility, typically 50 to 60% loss in reduction in area, Tables 3.1.1.1 and 3.1.1.2. As for most austenitic stainless steels, strength of A-286 and JBK-75 is relatively unaffected by both internal and external hydrogen.

Tensile ductility of JBK-75 with internal hydrogen is reduced at room temperature but is relatively little affected at lower temperature, tensile properties are provided in Table 3.1.1.3 and Figure 3.1.1.1 from room temperature to 77!K. Near room temperature JBK-75 with internal hydrogen exhibits very little ductility after necking begins, but ductility is greater at both lower temperature and elevated temperature. This is shown in Figure 3.1.1.2 for two sets of data, the lower curve represents the relative reduction in area after necking (RRA\*) [16], while the upper curve is the RRA as typically reported from total plastic strain for data from Table 3.1.1.3. In all cases the ductility and evidence of ductile fracture processes increase at lower temperature [16, 20].

Unlike other stainless steels in the presence of hydrogen, ductility in JBK-75 is not recovered at elevated strain rate up to 0.06ls<sup>-1</sup>, Figure 3.1.1.3; the data from Ref. [16] is given as the relative reduction in area after necking (RRA\*).

Aging tensile specimens after machining results in enhanced precipitation of the  $\eta$  phase due to surface deformation and a microstructure that is more sensitive to hydrogen, Table 3.1.1.4 [15], see also section 4.2.

# 3.1.2. Notched tensile properties

Notched tensile specimens show essentially no difference in properties when tested in helium or hydrogen at pressures up to 69!MPa, Table 3.1.2.1. The strength of notched tensile specimens of JBK-75 is unaffected by internal hydrogen for temperatures from 77!K to room temperature; the reduction in area of notched tensile specimens, however, is reduced somewhat at room temperature but relatively unaffected at low temperature, Figure 3.1.2.1.

In a separate study, A-286 was electrolytically precharged with internal hydrogen from a molten salt bath to various uniform hydrogen concentrations up to 40!wppm [25]. The notched tensile properties were then measured on single-edge-notched specimens. At a hydrogen concentration of 40!wppm, the notched tensile strength decreased by about 20% and the reduction in area decreased by 50%. The reported ductility loss near 25!wppm [25] is similar to that reported at room temperature for JBK-75 with internal hydrogen incorporated by thermal precharging from hydrogen gas as reported in Ref [20] (and shown in Figure 3.1.2.1).

## 3.2 Fracture mechanics

## 3.2.1 Fracture toughness

The fracture toughness of JBK-75 decreased by about half for material with high concentrations of internal hydrogen (>100!wppm), Table 3.2.1.1. Both ductile features and intergranular separation were observed on JBK-75 fracture surfaces [26]; however, uncharged materials primarily featured fracture modes consistent with ductile processes, while intergranular failure was more prevalent in specimens with internal hydrogen. Void nucleation was observed at grain boundaries, but evidence of ductile void formation was less in materials with greater volumes of grain boundary  $\eta$ -phase [26]. The  $\eta$ -phase was present on grain boundaries for all conditions tested, but the longer heat treatments resulted in greater volumes of  $\eta$ -phase, especially at the grain boundaries, and lower fracture toughness for materials with and without internal hydrogen [26].

Fracture toughness was determined [27] from 25.4!mm (1 in) thick, wedge open loading (WOL) specimens in constant displacement tests that did not meet plane strain requirements of standardized testing procedures [28]. These data are provided only as qualitative indicators since there is no other data reported in the literature for fracture toughness of A-286 or JBK-75 in external hydrogen gas. In 34.5!MPa gaseous helium, fracture toughness values ( $K_Q$ ) of 145 and 138!MPa!m<sup>1/2</sup> are reported at 295!K and 144!K respectively, while in 34.5!MPa gaseous hydrogen values of 100 and 152 MPa!m<sup>1/2</sup> are reported. The material for these tests was forged plate, heat W73 (Table 1.1.1), solution heat treated at 1255!K for 1 hour, oil-quenched and aged at 991!K for 16!hours followed by air-cooling.

#### **3.2.2 Threshold stress intensity factor**

Data from a number of austenitic stainless steels and iron-based (precipitation-strengthened) superalloys show that higher resistance to cracking under static loads in hydrogen generally corresponds to lower yield strength and similar values can be expected for a wide range of austenitic alloys [19]. Austenitic alloys with yield strengths less than about 700!MPa, in particular, have high resistance to cracking in high-pressure hydrogen gas environments under

#### A-286 Fe-25Ni-15Cr-2Ti-1.5Mn-1.3Mo-0.3V

static loads [19]. Threshold stress intensity factor ( $K_{TH}$ ) data for JBK-75 in high-pressure hydrogen gas, however, indicate that some microstructures are more susceptible than others, Table 3.2.2.1. Microstructure is especially important in two-phase alloy systems such as A-286, JBK-75 and other precipitation-strengthened alloys. In all cases, intergranular separation as well as ductile fracture processes were apparent from the fracture surfaces, with the fraction of ductile features scaling with threshold stress intensity factor. In addition, greater precipitation of  $\eta$ -phase in the grain boundary correlated with lower threshold [18]. The relatively low threshold stress intensity factor of the solution heat treated JBK-75 aged at the highest temperature is attributed to precipitation of  $\eta$ -phase at grain boundaries [18]. Compositional segregation in fusion-welded material also contributes to increased hydrogen susceptibility in these  $K_{TH}$  measurements [18, 19, 21].

In an earlier study, measurements of  $K_{TH}$  were attempted on 25.4!mm thick wedge open loading (WOL) specimens of A-286 that were not precracked and were loaded beyond the yield point [27]. None of the testing conditions satisfied the plane strain requirement of standardized testing procedures [28]. These data are provided for qualitative comparison. In 34.5!MPa gaseous hydrogen at room temperature, threshold stress intensity factor was found to be <113!MPa!m<sup>1/2</sup>. At 144!K, no crack propagation was observed at an applied stress intensity factor of 198!MPa!m<sup>1/2</sup> in 34.5!MPa gaseous hydrogen. The material for these tests was forged plate, heat W73 (Table 1.1.1), solution heat treated at 1255!K for 1 hour, oil-quenched and aged at 991!K for 16!hours followed by air cooling.

The effect of external hydrogen on crack growth in sustained loading of surface-flawed thin dogbone-like specimens of A-286 is reported to be negligible in 6.9!MPa gaseous hydrogen [29].

The threshold stress intensity factor for crack propagation of fatigue precracked A-286, with internal hydrogen from electrolytic precharging in molten salt, was measured as a function of hydrogen concentrations up to 30!wppm [30]. For these specimens, however, plane-stress conditions dominated thus the data cannot be compared to standardized plane-strain values of the stress intensity factor. Nevertheless, the relative  $K_{TH}$  (internal hydrogen relative to uncharged) was found to be about 0.75 for 30!wppm hydrogen [30]. In addition, the A-286 was less affected by hydrogen than most of the tested alloys including type 301 and 304 stainless steels and nickelbase superalloys (IN625 and IN718) [30].

# 3.3 Fatigue

Low-cycle fatigue experiments on A-286 show essentially no effect of hydrogen gas [31]. Hollow specimens pressurized to 34!MPa hydrogen and helium gas were tested in 1% total strain range and failed at approximately 2800 cycles.

# 3.4 Creep

Stress rupture tests in hydrogen gas at 922!K and a stress of 390!MPa result in a reduction in lifetime of about 20% for A-286, from 264 hours in 3.4!MPa air to 215!hours in 3.4!MPa hydrogen gas [31]. A large variation is associated with the rupture times in hydrogen.

#### 3.5 Impact

Charpy impact tests on EB-welded JBK-75 joints show some sensitivity to internal hydrogen (by thermal precharging from hydrogen gas) [32], see section 4.3.

#### **3.6 Disk rupture tests**

Disk rupture tests at room temperature show that A-286 is unaffected by pressurized hydrogen [33, 34]. Low-cycle fatigue in the disk rupture configuration (40lcycles to 0.5 of the rupture pressure) also did not affect the rupture pressure in hydrogen [34]. In a later report, disk rupture tests on JBK-75 and A-286 equivalent alloys showed considerable hydrogen embrittlement except in the solution heat-treated condition [35]. Rupture pressures for hydrogen were almost half of the rupture pressures in helium and evidence of intergranular failure modes were explained by  $\eta$ -phase precipitation at grain boundaries.

At elevated temperatures (360 to 700!K), hydrogen gas reduces the rupture pressure compared to helium gas. Exposure to hydrogen gas at 8.6!MPa for 48!hours further reduces the rupture pressure when pressurized by hydrogen gas [33]. This data underscores the importance of delayed effects due to hydrogen uptake and diffusion in metals.

#### 4. Metallurgical Considerations

#### 4.1 Primary processing

Carbide and sulfide inclusions are believed to have a significant impact on the fracture toughness of JBK-75 in the presence of hydrogen [36]. Deformation and thermomechanical processing accelerate the aging response of A-286 and JBK-75 [15, 17].

#### 4.2 Heat treatment

Since A-286 and alloys based on A-286 are precipitation-strengthened, the heat treatment is of primary importance for controlling microstructure and therefore, controlling strength and susceptibility to hydrogen embrittlement. Lower aging temperatures may help control precipitation kinetics and reduce the formation of undesirable phases such the  $\eta$ -phase [17]. The typical aging cycle for A-286 is 16 hours at 993!K. A two-step aging process is often employed for JBK-75: 8 hours at 948!K, followed by 8 hours at 873!K.

A-286 and JBK-75 in the solution heat-treated condition show little ductility loss in tensile tests with internal hydrogen, Figure 4.2.1. Aging results in a significant reduction in ductility due to  $\eta$ -phase precipitation and this reduction is exacerbated in the presence of internal hydrogen [3, 17]. This ductility loss is essentially independent of aging times greater than a few hours, although the volume fraction of  $\eta$ -phase increases substantially as does the fraction of intergranular failure [3].

Deformation induced by machining has been shown to accelerate  $\eta$ -phase precipitation in A-286, leading to a microstructure that is more susceptible to hydrogen embrittlement, Table 3.1.1.4 [15]. Similarly, intentional cold work accelerates the aging response of JBK-75 and results in ductility loss for aging times as short as one hour with internal hydrogen, Figure 4.2.1. These

data indicate that substantially shorter aging times may offer improved hydrogen compatibility without significant compromise on strength, and that standard aging cycles (993K for 16 hours) are not appropriate for thermomechanically processed materials for service in hydrogen. Values of fracture toughness [26], Table 3.2.1.1, and threshold stress intensity factor in gaseous hydrogen [19], Table 3.2.2.1, also support the principle that shorter aging times and lower temperatures result in improved hydrogen compatibility.

# 4.3 Properties of welds

Tensile testing of JBK-75 gas tungsten arc (GTA) welds with high concentrations of internal hydrogen show significant losses in ductility [21]. Interdentritic regions in these welds are rich in titanium and nickel, and thus believed to be preferential sites for precipitation of the  $\eta$ -phase (Ni<sub>3</sub>Ti) and vulnerable to intergranular fracture [6, 17, 18]. Fracture of the welds was primarily by microvoid coalescence with some evidence of the underlying weld microstructure, however, localized regions of intergranular fracture were observed near the surface of specimens with internal hydrogen, i.e. in regions where the hydrogen concentration was greatest. As in base material [6], the dimple size was reduced in the presence of hydrogen, presumably due to increased activation of nucleation sites, for example in the interdentritic regions. The tensile properties of GTA welds are listed in Table 4.3.1. These data are shown for reference only as they represent the properties of a composite specimen (fusion zone, heat-affected zone and base metal), however, they do demonstrate the effect of hydrogen on the ductility of the welds.

The threshold stress intensity factor of a fusion weld of JBK-75 in hydrogen was reported to be about half that measured for similarly aged forged base metal [19], Table 3.2.2.1. The increased susceptibility is attributed to the macrosegregation inherent to fusion welding processes.

Like the single-phase austenitic stainless steels, the susceptibility to hydrogen embrittlement (as measured by tensile ductility) of JBK-75 electron-beam (EB) welded joints reaches a minimum near room temperature for material with internal hydrogen [32], Table 4.3.2 and Figure 4.3.1. Charpy impact tests, however, show the greatest susceptibility to hydrogen embrittlement at lower temperature and only a nominal effect at room temperature [32]. Overaging these welded joints (30!h at 1013!K) increases susceptibility to hydrogen embrittlement, due to  $\eta$ -phase precipitation [32].

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# **Austenitic Stainless Steels**

#### A-286 Fe-25Ni-15Cr-2Ti-1.5Mn-1.3Mo-0.3V

heat	Fe	Cr	Ni	Ti	Mn	Мо	V	Al	Si	С	В	other	Ref.
UNS S66286	Bal	13.50 16.00	24.0 27.0	1.90 2.35	2.00 max	1.00 1.50	0.10 0.50	0.35 max	1.00 max	0.08 max	0.0010 0.010	0.40 max P; 0.030 max S	[37]
W69	Bal	15.07	25.58	1.93	1.47	1.35	0.30	0.13	0.61	0.052	0.0055	0.019!P; 0.010!S	[10]
W73	Bal	14.15	24.88	2.21	1.20	1.25	0.22	0.16	0.63	0.048	0.47	0.010 S; 0.016!P; 0.01 Zr	[27]
P81	Bal	14.0	24.33	2.15	0.13	1.16			0.16	0.054			[19]
B93	Bal	14.90	24.93	2.15	1.32	1.25	0.21	0.19	0.63	0.068	0.004	0.003!S; 0.018!P	[3]
V96	Bal	14.02	24.38	2.09	0.28	1.37	0.2	0.13	0.22	0.024	0.0046	0.1!Cu; 0.08!Co; 0.001!S; 0.001!P	[14]
V-57	Bal	14.8	26.0	3.0	0.3	1.25	0.3	0.25	0.6	0.05	0.01	Nominal values for alloy V-57	[11]

Table 1.1.1. Specification limits for A-286 and composition of several heats of A-286 stainless steel used to study hydrogen effects.

#### **Austenitic Stainless Steels**

#### A-286 Fe-25Ni-15Cr-2Ti-1.5Mn-1.3Mo-0.3V

heat	Fe	Cr	Ni	Ti	Mn	Мо	V	Al	Si	C	В	other	Ref.
UNS	Dol	13.50	29.00	2.0	0.20	1.00	0.10	0.15	0.10	0.01	0.002	0.006 max S;	
S66286	Dal	16.00	31.00	2.3	max	1.50	0.50	0.35	max	0.03	max	0.010 max P	
T75	Bal	14.48	30.46	2.07	0.11	1.22	0.25	0.27	0.15	0.020	0.0010		[6]
B80	Bal	14.02	29.58	2.10	<0.01	1.28	0.35	0.16	< 0.01	0.019	< 0.001		[15]
O80	Bal	15.3	29.8	2.1	0.011	1.2	0.42	0.3	0.075	0.012	0.0011	0.004!S; 0.01!P	[17]
P80	Bal	15.5	30.7	2.1	0.053	1.2	0.26	0.2	0.032	0.017	<0.0005	0.0013!S; <0.002!P	[19]
B83w	Bal	15.0	30.0	2.2	0.1	1.2		0.2	0.1	0.03	0.001	0.01!S; 0.01!P	[21]
X93	Bal	15.22	29.48	1.85	0.19	1.53	0.26	0.20	0.17	0.024	0.0019	0.004 S; 0.011!P	[22]

Table 1.1.2. Specification limits for JBK-75 and composition of several heats of JBK-75 stainless steel used to study hydrogen effects.

w = composition of the weld fusion zone

	Temperature	Dressure	$\Phi = \Phi_o \exp(-E$	$_{\Phi}/RT$	$S = S_o \exp(-E)$		
Material	Range (K)	Range (MPa)	$\frac{\Phi_o}{\left(\frac{\mathrm{mol}\ \mathrm{H}_2}{\mathrm{m}\cdot\mathrm{s}\cdot\sqrt{\mathrm{MPa}}}\right)}$	$\frac{E_{\Phi}}{\left(\frac{\mathrm{kJ}}{\mathrm{mol}}\right)}$	$\frac{S_o}{\left(\frac{\text{mol } \text{H}_2}{\text{m}^3 \cdot \sqrt{\text{MPa}}}\right)}$	$\frac{E_s}{\left(\frac{\mathrm{kJ}}{\mathrm{mol}}\right)}$	Ref.
JBK-75, heat X93	483-703	0.1	4.36 x 10 <sup>-4</sup>	62.10	145	13.58	[22]
Average of several austenitic alloys †	423-700	0.1-0.3	1.2 x 10 <sup>-4</sup>	59.8	179	5.9	[24]
Average of six austenitic alloys	473-703	0.1	2.81 x 10 <sup>-4</sup>	62.27	488	8.65	[38]
Average of four austenitic alloys	373-623	$1 \times 10^{-4} - 0.03$	5.35 x 10 <sup>-5</sup>	56.1	266	6.86	[39]

Table 2.1. Permeability and solubility relationships for JBK-75 and average relationships determined for several austenitic stainless steels.

<sup>†</sup> Data from Ref. [24] is determined for deuterium: permeability has been corrected here to give permeability of hydrogen (by multiplying by the square root of the mass ratio:  $\sqrt{2}$ ); solubility is assumed to be independent of isotope.

Table 2.2. Hydrogen concentration of modified A-286 alloys measured using hot extraction after thermal precharging in hydrogen gas.

Material	Surface condition	Thermal	Hydro concent	Ref.	
		precharging	wppm	appm	
JBK-75 ST + A		69"MPa H <sub>2</sub> 573"K	54	3000	[16]
"modified A-286"	600 grit finish		80	4500	
Annealed	Electropolished	69"MPa H <sub>2</sub>	81	4500	[22]
"modified A-286"	600 grit finish	470"K	51	2900	[23]
HERF	Electropolished		55	3100	
JBK-75 ST + A		10"MPa H <sub>2</sub> 573"K	25	1400	[20]

HERF = high energy rate forging, ST = solution treatment, A = age

Table 3.1.1.1. Smooth tensile properties of A-286 stainless steel at room temperature; measured in external hydrogen gas or with internal hydrogen (measured in air after thermal precharging in hydrogen gas).

Material	Thermal precharging	Test environment	Strain rate (s <sup>-1</sup> )	S <sub>y</sub> (MPa)	S <sub>u</sub> (MPa)	El <sub>u</sub> (%)	El <sub>t</sub> (%)	RA (%)	Ref.	
A 286	None	69"MPa He		724	1117		26	47	[10]	
A-280	None	69"MPa H <sub>2</sub>		710	1131		34	49	[12]	
A-286, heat W69	None	69"MPa He		848	1089		26	44	5.1.0	
ST + A (1173K/2h + 993K/16h)	None	69"MPa H <sub>2</sub>	0.67 x 10 <sup>-3</sup>		1117		29	43	[10, 31]	
A-286	None	Air		760†	1065		21	32	5.6	
А	None	69"MPa H <sub>2</sub>				(R	$(RRA \sim 1)$		[6, 9]	
(990K/16h)	(1)	Air				(RI	$RA \sim 0$	0.5)	~1	
A-286	None	Air		850†	1105			(30)		
HERF + A	None	69"MPa H <sub>2</sub>				(R	(RRA ~ 1)		[9]	
(990K/16h)	(1)	Air				$(RRA \sim 0.5)$				
A-286	None	Air		440†	750			(58)	[0]	
HERF	(1)	Air				(R	RRA ~	1)	[7]	
A-286, heat V96	None	34"MPa He	0.0	843	1166	24		50		
ST + A (1266K/1h/WQ+ 994K/16h/AC)	None	34"MPa H <sub>2</sub>	8.3 x 10 <sup>-6</sup>	839	1159	24		51	[14]	
V-57, heat V-57	None	Air		690	1145		32	50		
ST + A	None	69"MPa H <sub>2</sub>				(RF	$RA \sim 0$	.95)	[11]	
990K/16h/AC)	(3)	Air				(RR	$A \sim 0$	).25)		

Values in parenthesis are determined from plots.

HERF = high energy rate forging, ST = solution treatment, A = age, WQ = water quench,

OQ"="oil quench, AC = air cool

† stress at 0.2% strain

(1) 69 MPa hydrogen, 475"K, 1500 h

(2) 24"MPa hydrogen, 475"K, 400"h (gauge diameter"="6.5"mm diameter)

Table 3.1.1.2. Smooth tensile properties of JBK-75 stainless steel at room temperature; measured in external hydrogen gas, or with internal hydrogen (measured in air after thermal precharging in hydrogen gas).

Material	Thermal precharging	Test environment	Strain rate† (s <sup>-1</sup> )	S <sub>y</sub> (MPa)	S <sub>u</sub> (MPa)	El <sub>u</sub> (%)	El <sub>t</sub> (%)	RA (%)	Ref.
JBK-75, heat T75	None	Air		875	1305		21	55	
ST + A (1200K/2h +	None	69"MPa H <sub>2</sub>				R	RA ~	1	[6]
990K/16h/AC)	(1)	air				RF	$RRA \sim 0.9$		
JBK-75, heat P80	None	Air	0.021	717	1131		28	51	
A (993K/16h)	None	172"MPa H <sub>2</sub>	mm/s†					47	[13]
JBK-75, heat B80	None	Air	0.33	702	1105	18	23	45	
A (993K/16h)	(2)	Air	x 10 <sup>-3</sup>	703	1100	16	17	20	[15]
JBK-75, heat O80	None	Air		716	1130	22		51	
ST + A (1253K/1h/WQ + 993K/16h)	(3)	172"MPa H <sub>2</sub>	0.83 x10 <sup>-3</sup>	723	1137	16		24	[17]
JBK-75, heat O80	None	Air	0.83	1083	1302	11		45	
8% CW + A (948K/8h)	(3)	172"MPa H <sub>2</sub>	x10 <sup>-3</sup>	1089	1295	12		18	[17]
JBK-75	None	Air		763	1109		29	58	
ST + A (1253K/1h/WQ + 1013K/8"h)	(4)	Air	0.017 mm/s†	763	1110		26	43	[20]
JBK-75	None	Air	0.017	759	1090		32	59	
A (1013"K/8"h)	(5)	Air	mm/s†	745	1071		31	40	[32]

<sup>†</sup> when strain rate is not known, displacement rates are quoted if reported

ST = solution treatment, A = age, WQ = water quench, CW = cold work (diameter reduction)

(1) 24"MPa hydrogen gas, 475"K, 100"h (gauge diameter"="3"mm); calculated concentration gradient of 45 to 4 wppm hydrogen surface to center (2500 to 250 appm)

(2) 69 MPa hydrogen gas, 473"K, 158"h (gauge diameter"="5"mm); calculated concentration gradient of 45 to 9"wppm hydrogen surface to center (2500 to 500 appm); however vacuum extraction indicated hydrogen concentration of about 20% higher

(3) 69 MPa hydrogen gas, 473"K, 240"h (gauge diameter"="5"mm); calculated concentration gradient of 99 to 2"wppm hydrogen surface to center (5500 to 100 appm)

(4) 10"MPa hydrogen gas, 573"K, 340"h (gauge diameter ="5"mm); 25"wppm hydrogen (1400"appm) measured by ion-microprobe sectional hydrogen analysis

(5) 10"MPa hydrogen gas, 573"K, 340 h (gauge thickness"="2"mm); 25"wppm hydrogen (1400"appm) measured by ion-microprobe sectional hydrogen analysis

#### A-286 Fe-25Ni-15Cr-2Ti-1.5Mn-1.3Mo-0.3V

Table 3.1.1.3. Smooth tensile properties of JBK-75 stainless steel as a function of temperature	;
with internal hydrogen (measured in air after thermal precharging in hydrogen gas).	

	0			0	0	0	0 /		
Material	Thermal precharging	Test environment	Strain rate† (s <sup>-1</sup> )	S <sub>y</sub> (MPa)	S <sub>u</sub> (MPa)	El <sub>u</sub> (%)	El <sub>t</sub> (%)	RA (%)	Ref.
	None	Air 203 K		763	1109		28.9	58.1	
	(1)	All 293 K		763	1110		26.1	43.4	
JBK-75	None	A ; 222"V		778	1152		30.2	57.7	
ST + A	(1)	All 223 K	0.17	775	1153		29.6	51.4	[20]
(1253K/1h/WQ	None	Air 152"V	mm/s†	806	1190		31.3	57.3	[20]
+ 1013K/8h)	(1)	All 155 K		793	1207		33.1	56.3	
	None	1. in 77 W		876	1412		41.6	60	
	(1)			868	1417		41.6	59.2	

<sup>†</sup> when strain rate is not known, displacement rates are quoted if reported

(1) 10"MPa hydrogen gas, 573"K, 340 h (gauge diameter"="5"mm); 25"wppm uniform hydrogen (1400"appm)

Table 3.1.1.4. Smooth tensile properties of JBK-75 stainless steel at room temperature as function of surface deformation due to machining; with internal hydrogen (measured in air after thermal precharging in hydrogen gas).

Material	Thermal precharging	Test environment	Strain rate (s <sup>-1</sup> )	S <sub>y</sub> (MPa)	S <sub>u</sub> (MPa)	El <sub>u</sub> (%)	El <sub>t</sub> (%)	RA (%)	Ref.
JKB-75, heat B80	None	Air		702	1105	18.2	23.4	45.3	
Age† + machine	(1)	Air		703	1100	16.5	16.7	20.2	
JBK-75, heat B80	None	Air	0 33	702	1121	18.6	23.6	49.0	
Age† + machine + grind	(1)	Air	x 10 <sup>-3</sup>	716	1106	15.8	16.2	23.8	[15]
JBK-75, heat B80	None	Air		806	1124	18.6	23.7	46.5	
Machine + age† + grind	(1)	Air		805	1090	11.7	12.0	14.7	

† 993K/16h

(1) 69 MPa hydrogen gas, 473"K, 158"h (gauge diameter"="5"mm); calculated concentration gradient of 45 to 9"wppm hydrogen surface to center (2500 to 500 appm); however vacuum extraction indicated hydrogen concentration about 20% higher.

Material	Specimen	Thermal precharging	Test environment	Displ. rate (mm/s)	S <sub>y</sub> † (MPa)	σ <sub>s</sub> (MPa)	RA (%)	Ref.
A-286, heat W69		None	69"MPa He		848	1606	5.6	
ST + A	(2)			0.7				[10,
(1173K/2h/WQ+	(a)	None	69"MPa H <sub>2</sub>	x10 <sup>-3</sup>		1565	6.2	31]
993K/16h/AC)			2					
A-286, heat V96		None	34"MPa He		843	1826		
ST + A	(b)			0.21				[1/]
(1266K/1h/WQ+		None	34"MPa H <sub>2</sub>	x10 <sup>-3</sup>	839	1756		[14]
994K/16h/AC)			2					

Table 3.1.2.1. Notched tensile properties of A-286 stainless steel at room temperature; measured in external hydrogen gas.

ST = solution treatment, A = age, WQ = water quench, AC = air cool

† yield strength of smooth tensile specimen

(a) V-notched specimen: 60° included angle; minimum diameter = 3.81"mm; maximum diameter = 7.77"mm; notch root radius = 0.024"mm. Stress concentration factor (K<sub>t</sub>)"="8.4.

(b) Notch (minimum) diameter = 6.35"mm. Stress concentration factor (K<sub>t</sub>) = 6.0.

Material	Test method	Thermal precharging	Test environment	S <sub>y</sub> (MPa)	$egin{array}{c} K_Q^{\dagger} \ (MPa \ m^{1/2}) \end{array}$	Ref.
JBK-75, heat B80	WOL	None	Air	717	139	
ST + A	J-integral	1 tone	7 111	/1/	157	[36,
(1253K/1h/WQ +	3PB	(1)	Air	723	77	40]
993K/16h)	J-integral	(1)	All	125	11	
JBK-75, heat O80		None	Air	937	99	
HERF + A	3PB					[26]
(1253K; 948K/8h +	J-integral	(1)	Air		44	[20]
873K/8h)		~ /				
JBK-75, heat O80	200	None	Air	960	89	
HERF + A	JFD Lintagral	(1)			4.1	[26]
(1253K; 948K/32h)	J-Integral	(1)	Aır		41	
JBK-75, heat O80	3DD	None	Air	964	87	
HERF + A	JEEM	(1)			25	[26]
(1253K; 948K/96h)	LEFIVI	(1)	Aır		35	

Table 3.2.1.1. Fracture toughness of JBK-75 stainless steel at room temperature; measured in air with internal hydrogen (thermal precharging in hydrogen gas).

HERF = high energy rate forging, A = age, ST = solution treatment, WQ = water quench, WOL = wedge open loading specimen, 3PB = 3-point bending specimen, LEFM = linear elastic fracture mechanics

† not clear if plane strain requirements are met in these studies

 (1) 138"MPa hydrogen, 573"K, 1500"h; estimated uniform hydrogen concentration of 120-140 wppm (6700 - 8000 appm) [36, 40]

Table 3.2.2.1. Threshold stress intensity factor for A-286 and JBK-75; measured in external hydrogen gas. The testing procedure is believed to have satisfied the requirements of ASTM E 1681-99 [28].

Material	S <sub>y</sub> †	RA †	Threshold Stress (MPa	Intensity Factor $m^{1/2}$ )	Ref.
	(MPa)	(%)	100 MPa H <sub>2</sub>	200 MPa H <sub>2</sub>	
A-286, heat P81					
ST +A	779	46		94*	[19] ‡
(1253 K/1 h/WQ + 993 K/16 h)					
JBK-75, heat P80					
ST + A	717	51	44	47	[19] ‡
(1253 K/1 h/WQ + 993 K/16 h)					
JBK-75, heat P80					
HERF + A	855	37	109*	116*	[10] +
(1243K/WQ + 948K/8h +	055	51	107	110	[17] +
873K/8h)					
JBK-75, heat P80					
HERF + A	923	38	69	66	[19] ‡
(1243K/WQ + 948K/32h)					
JBK-75, heat P80				50	
Fusion weld + A	~700		$\sim$	not reported)	[19] ‡
(948K/8h + 873K/8h)			$(\Pi_2 \text{ pressure})$		

HERF = high-energy rate forging, ST = solution treatment, A = age, WQ = water quench

\* did not satisfy plane strain requirements for analysis of linear elastic fracture mechanics

† yield strength and reduction in area of smooth tensile specimen, not exposed to hydrogen

‡ data also reported in Ref. [13, 18, 41]

Table 4.2.1. Smooth tensile properties of JBK-75 stainless steel at room temperature as a function of aging time and cold-work; measured in external hydrogen gas with internal hydrogen (thermal precharging in hydrogen gas).

Condition	Aging	Thermal	Test	Strain	Sy	S <sub>u</sub>	El <sub>u</sub>	Elt	RA	Ref.
	time	precharging	environment	rate	(MPa)	(MPa)	(%)	(%)	(%)	
JBK-75, heat O80	ST	None	Aır		241	620	35		70	. [17]
		(1)	172"MPa H <sub>2</sub>		245	618	34		67	
	4 h	None	Air		565	1058	26		61	
		(1)	172"MPa H <sub>2</sub>		560	1012	18		24	
ST + A (1253K/	8 h	None	Air	0.83 x 10 <sup>-3</sup> s <sup>-1</sup>	632	1091	24		57	
		(1)	172"MPa H <sub>2</sub>		640	1063	16		23	
	12 h	None	Air		672	1131	22		51	
993K)		(1)	172"MPa H <sub>2</sub>		683	1092	16		21	
	16 h	None	Air		716	1130	22		51	
		(1)	172"MPa H <sub>2</sub>		723	1137	16		24	
JBK-75, heat O80	1 h	None	Air		987	1169	13		60	
		(1)	172"MPa H <sub>2</sub>		1054	1216	12		25	
8% CW +	4 h	None	Air		1100	1288	11		52	
		(1)	172"MPa H <sub>2</sub>		1136	1282	10		23	
(948"K)	8 h	None	Air		1083	1302	11		45	
		(1)	172"MPa H <sub>2</sub>		1089	1295	12		18	
JBK-75, heat O80	1 h	None	Air		1196	1306	6.9		54	
		(1)	172"MPa H <sub>2</sub>		1226	1325	7.8		31	
2007 CW .	4 h	None	Air		1178	1340	8.5		45	
20% C w +		(1)	172"MPa H <sub>2</sub>		1192	1326	9.2		22	
(948K)	8 h	None	Air		1085	1304	9.5		40	
		(1)	172"MPa H <sub>2</sub>		1123	1295	9.6		19	
JBK-75, heat O80	1 h	None	Air		1212	1337	5.8		50	
		(1)	172"MPa H <sub>2</sub>		1240	1350	7.2		21	
36% CW +	4 h	None	Air		1029	1269	10		44	
		(1)	172"MPa H <sub>2</sub>		1075	1268	9.5		19	
	8 h	None	Air		785	1169	15		48	
(948K)		(1)	172"MPa H <sub>2</sub>		878	1152	12		20	

ST = solution treatment, A = age, WQ = water quench; CW = cold work (diameter reduction) (1) 69 MPa hydrogen gas, 473K, 240<sup>th</sup> (gauge diameter"="5"mm): calculated concentration

gradient of approximately 99 to 2"wppm hydrogen surface to center (5500 to 100 appm)

#### A-286 Fe-25Ni-15Cr-2Ti-1.5Mn-1.3Mo-0.3V

Table 4.3.1. Smooth tensile properties of JBK-75 composite GTA weld specimens at room temperature; with internal hydrogen (measured in air after thermal precharging in hydrogen gas), or measured in external hydrogen gas with internal hydrogen (thermal precharging in hydrogen gas).

Material	Thermal precharging	Test environment	Strain rate (s <sup>-1</sup> )	S <sub>y</sub> (MPa)	S <sub>u</sub> (MPa)	El <sub>u</sub> (%)	El <sub>t</sub> (%)	RA (%)	Ref.
JBK-75, heat B83w†	None	Air	0 33	781	1014	6.0	8.2	38	
	(1)	Air		749	980	4.6	4.8	24	
Aged	(2)	Air	x 10 <sup>-3</sup>	796	993	4.2	4.4	22	[21]
(948K/8h + 873K/8h)	(2)	172MPa H <sub>2</sub>		760	953	4.6	5.0	23	

<sup>†</sup> The base material for these studies was HERF (high energy rate forging), back extrusions of JBK-75, machined to cylindrical shape (10"cm diameter, 1.5"cm wall thickness) with circumferential double J grooves; eight to ten weld passes were required to fill groove. The filler material was also JBK-75 matched to the composition of the base metal. Tensile bars contain base material and heat affected zone with the fusion zone centered in the gauge length, and were aged after machining.

- (1) 24"MPa hydrogen gas, 473"K, 240"h (gauge diameter"="5"mm): calculated concentration gradient of 45 to 4 wppm surface to center (2500 to 200"appm)
- (2) 69"MPa hydrogen gas, 473"K, 240"h (gauge diameter"="5"mm): calculated concentration gradient of 72 to 7 wppm surface to center (4000 to 400"appm)

Material	Thermal precharging	Test temperature (K)	Strain rate† (s <sup>-1</sup> )	S <sub>y</sub> (MPa)	S <sub>u</sub> (MPa)	El <sub>u</sub> (%)	El <sub>t</sub> (%)	RA (%)	Ref.
JBK-75	None	293	0.017 mm/s†	759	1090		32	59	[32]
Aged (1013K/8h)	(1)			745	1071		31	40	
JBK-75 EB welds Aged (1013K/8h)	None	293		800	1041		18	52	
	(1)			784	1032		16	33	
	None	193		826	1096		19	51	
	(1)			832	1121		20	44	
	None	77		909	1306		25	50	
	(1)			921	1318		24	41	

Table 4.3.2. Smooth tensile properties of JBK-75 EB-weld specimens at low temperatures; with internal hydrogen (measured in air after thermal precharging in hydrogen gas).

EB = electron beam

† when strain rate is not known, displacement rates are quoted if reported

 10"MPa hydrogen gas, 573"K, 340"h (gauge thickness"="2"mm); 25"wppm hydrogen (1400"appm) in the base metal and 16"wppm (920"appm) in the weld metal, measured by ion-microprobe sectional hydrogen analysis



Figure 2.1. Permeability in JBK-75 and average relationships determined for several austenitic stainless steels. Data from Ref. [24] is determined for deuterium: permeability has been corrected here to give permeability of hydrogen by multiplying by the square root of the mass ratio ( $\sqrt{2}$ ).



Figure 2.2. Solubility in JBK-75 and average relationships determined for several austenitic stainless steels. Data from Ref. [24] is determined for deuterium; however, solubility is assumed to be independent of hydrogen isotope.



Figure 3.1.1.1. Smooth tensile properties of JBK-75 stainless steel as a function of temperature; with internal hydrogen (measured in air after thermal precharging in hydrogen gas). Data also presented in Table 3.1.1.3. [20]



Figure 3.1.1.2. Relative reduction of area (RRA) of smooth tensile specimens of JBK-75 stainless steel as a function of temperature; with internal hydrogen (measured in air after thermal precharging in hydrogen gas). Data from Ref. [20] is also presented in Table 3.1.1.3 and Figure 3.1.1.1.



Figure 3.1.1.3. Relative reduction in area of smooth tensile specimens of JBK-75 stainless steel at room temperature as a function of strain rate.



Figure 3.1.2.1. Notched tensile properties of JBK-75 stainless steel as a function of test temperature; measured in air with internal hydrogen (thermal precharging in hydrogen gas). [20]



Figure 4.2.1. Relative reduction of area (RRA) as a function of aging time for several microstructural conditions of JBK-75; measured in external (172"MPa)  $H_2$  gas with internal hydrogen (heat O80), data also reported in Table 4.2.1, and for A-286 with internal hydrogen (heat B93). ST = solution treatment, A = age, WQ = water quench, CW = cold work



Figure 4.3.1. Reduction in area of JBK-75 stainless steel EB-welded joints as a function of test temperature; measured in air with internal hydrogen (thermal precharging in hydrogen gas). [32] EB = electron beam, A = age