

MECHANICAL TESTING OF CARBON STEEL IN HIGH PRESSURE HYDROGEN (U)

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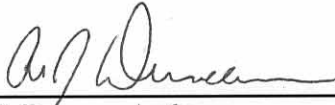
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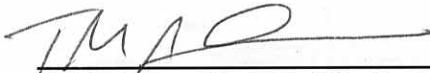
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1.0 EXECUTIVE SUMMARY

The methods and interim results from a testing program to quantify hydrogen effects on mechanical properties of carbon steel pipeline and pipeline weld materials are provided. The scope is carbon steels commonly used for natural gas pipelines in the United States that are candidates for hydrogen service in the hydrogen economy. The mechanical test results will be applied in future analyses to evaluate service life of the pipelines. The results are also envisioned to be part of the bases for construction codes and structural integrity demonstrations for hydrogen service pipeline and vessels.

Tensile properties of one type of steel (A106 Grade B) in base metal, welded and heat affected zone conditions were tested at room temperature in air and high pressure (1500 psig) hydrogen. A general reduction in the materials ability to plastically deform was noted in this material when specimens were tested in 1500 psig hydrogen. Furthermore, the primary mode of fracture was changed from ductile rupture in air to cleavage with secondary tearing in hydrogen.

The mechanical test program will continue with tests to quantify the fracture behavior in terms of J-R curves for these materials at air and hydrogen pressure conditions.

2.0 BACKGROUND

An infrastructure of new and existing pipelines and systems will be required to carry and to deliver hydrogen as an alternative energy source under the hydrogen economy. Carbon and low alloy steels of moderate strength are currently used in hydrogen delivery systems as well as in the existing natural gas systems. It is critical to understand the material response of these standard pipeline materials specified by the American Petroleum Institute (API) when they are subject to pressurized gases of pure hydrogen or its mixture with methane since hydrogen is well known in deteriorating the mechanical properties of steels.

A literature survey for existing mechanical property data on carbon and low alloy steels exposed to hydrogen gas was conducted to support the program led by the Concurrent Technologies Corporation for hydrogen pipeline life management. Data needs were identified certain materials. Among those identified, data for tests in hydrogen at a standard method was needed.

Tensile data for carbon steels in hydrogen atmospheres (100 ATM) was planned to develop a data set for transmission pipelines. Three pipe alloys were considered for the initial study. Alloys API X42, API X70 and ASTM A106 grade B were chosen because of their wide utilization in the pipeline industry. The compositional requirements from each specification are listed in Table I. Alloy A106 grade B was eventually chosen for the initial portion of the study because the particular pipe section available had a large wall thickness, which left ample material for the machining of specimens and could be sectioned with the entire gage section intersecting the base metal, heat affected zone or weld metal. This report documents the interim results from the testing of specimens machined from this welded pipe section.

Table I: Chemical Specification for Pipe Materials ^{1,2}

	Composition, %		
	A 106 Grade B	API X42 (welded)	API X70 (welded)
Carbon, max ^A	0.3	0.26	0.26
Manganese	0.29-1.06	1.3 (max)	1.65 (max)
Phosphorus, max	0.035	0.03	0.03
Sulfur, max	0.035	0.03	0.03
Silicon, min	0.1		
Chrome, max ^B	0.4		
Copper, max ^B	0.4		
Molybdenum, max ^B	0.15		
Nickel, max ^B	0.4		
Vanadium, max ^B	0.08		
Titanium, max		0.06	0.06

^A For each reduction of 0.01% below the specified carbon maximum, an increase of 0.06% manganese above the specified maximum will be permitted up to a maximum of 1.35%.

^B These five elements combined shall not exceed 1%.

3.0 EXPERIMENTAL

A section of 4" schedule XXH pipe (4.5 inches OD X 0.674 inches wall thickness) was circumferentially welded using welding procedure specification WPS-P1-TA.³ The multi-pass SMAW weld was performed with certification and without any post weld heat treatment. The root pass of the weld was performed by gas tungsten arc welding (GTAW) with filler rod ER70S-2 filler rod, while each remaining pass used shielded metal arc welding (SMAW) with E7018 filler rod. Figure 1 shows a picture of the pipe section after welding.

After the weld procedure was complete the pipe was sectioned in the using electro-discharge milling (EDM) and tensile specimens were machined in the L-C orientation (see Figure 2). Specimens were machined in a "dog bone" configuration from the pipe section with the gage section intersecting the base metal, weld metal and HAZ regions of the pipe (see Figure 3). The initial tests were performed at ambient temperatures and atmospheric conditions (see Table II). Also, additional samples were tested in 1500 psig research grade hydrogen gas (99.999% pure).

The specimens were tested at room temperature (i.e. 64 – 78°F) on a screw driven MTS Sintech Renew™ 1125 load frame with a 1000 lb load cell at a strain rate of 1×10^{-4} in/in/sec. The load frame is located inside a hydrogen environmental test facility at SRNL which utilizes engineering controls to ensure flammable gas concentrations in the facility are maintained below the lower flammability limit and engineered barriers to protect personnel from an explosion. The data was collected using MTS Testworks™ v3.1 data acquisition software.

The specimens that were tested in hydrogen were loaded into a high-pressure vessel manufactured by Autoclave Engineering™ that is equipped with a translational feedthrough. The chamber was constrained in the load frame (to prevent premature loading of the specimen), purged with inert gas several times and pressurized to 1500 psig. The soak time in hydrogen gas, at pressure, was 30 minutes. The pressure was controlled remotely using an independent control system utilizing LabView™ v5.0 software. A picture of the test set up is shown in Figure 4.

The tests were performed to failure and the specimens were examined after the test to measure reduction in area, to verify plastic strain to failure and to characterize the fracture surface. Optical microscopy and scanning electron microscopy were utilized to accomplish this analysis. The raw data of load and crosshead deflection were analyzed using a technique to compensate for machine compliance and slippage in the grip section. The procedure for this correction is described in a study by Lam *et al.*⁴. Engineering stress and strain were calculated and plotted in the results and discussion.



Figure 1: Photograph of Alloy A 106 Grade B Pipe Section after Welding

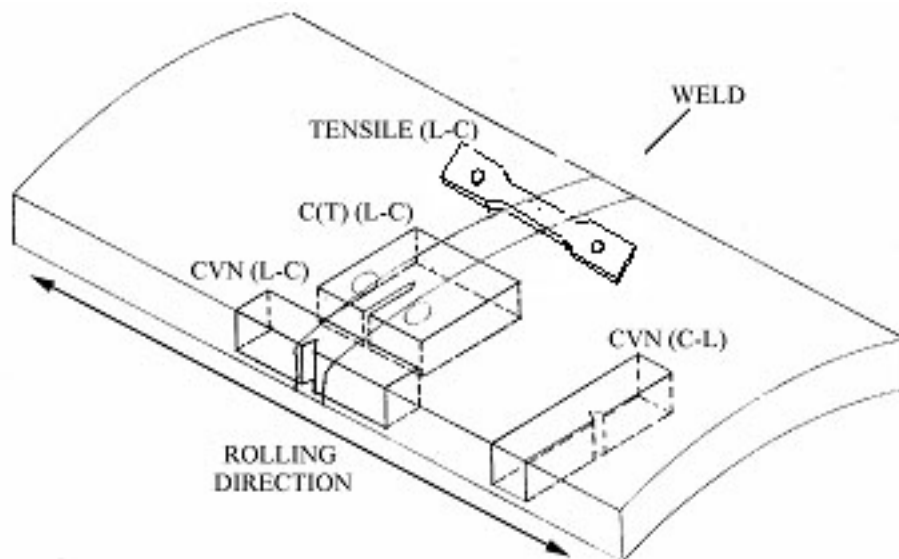
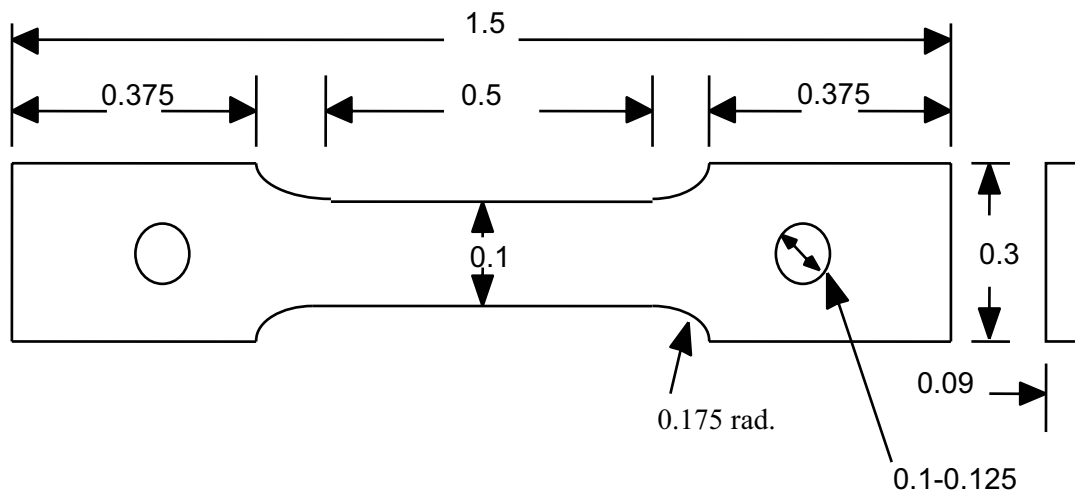


Figure 2: Schematic of Pipe Section illustrating the Orientation of Tensile Specimens



All dimensions in inches

Figure 3: Schematic and Dimensions of Dog bone Tensile Specimen



Figure 4: Hydrogen Test Load Frame Set Up and Configuration

Table II: Test Matrix for Experimental Program

106 Grade B	Sample	1 ATM (Air)	100 ATM (H₂)
Base Metal	1	106B001A1	106B100H1
	2	106B001A3	106B100H2
	3	106B001A5	106B100H3
	4	106B001A7	106B100H4
	5	106B001A8	106B100H5
	6	106B001A9	106B100H6
Weld Metal	1	106W001A1	106W100H1
	2	106W001A2	106W100H2
	3	106W001A4	106W100H3
	4	106W001A6	106W100H4
	5	106W001A7	106W100H5
	6	106W001A8	106W100H6
HAZ	1	106H001A1	106H100H1
	2	106H001A2	106H100H2
	3	106H001A3	106H100H3
	4	106H001A4	106H100H4
	5	106H001A5	106H100H5
	6	106H001A6	106H100H6

4.0 RESULTS AND DISCUSSION

The changes resulting from exposure of carbon steels to hydrogen environments is well known. As stated in the review of the related open literature⁵,

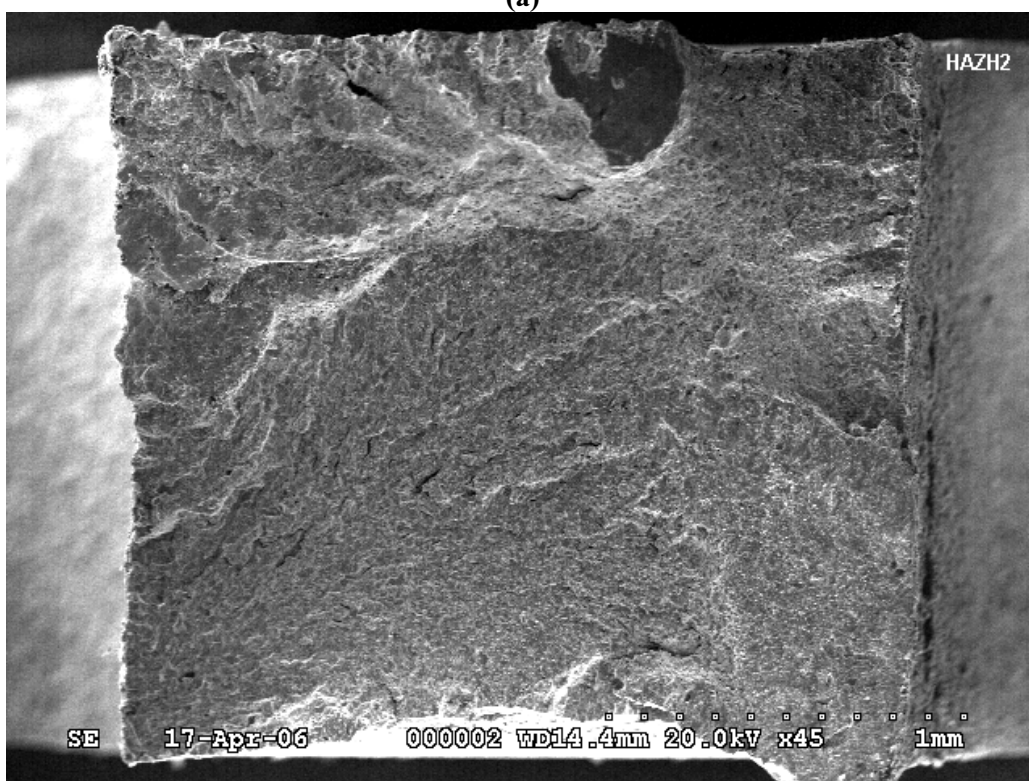
“...the deformation capacity (ductility), fracture mechanics properties including fracture toughness and fatigue crack propagation characteristics are deteriorated as the hydrogen pressure increases.”

The behavior of this “deformation capacity” is illustrated when the fracture surfaces of tensile specimens are examined for specimens tested in air versus hydrogen. For example, Figure 5 (a and b) shows the fracture surfaces for the HAZ tensile specimens tested in air and 1500 psig hydrogen at low magnification. Figure 6 (a and b) shows the fracture surfaces for the same HAZ tensile specimens at higher magnification. From these figures, the effects of hydrogen on the deformation capacity can be readily observed. Specifically, the overall “reduction in area” in the necked region is significantly greater in the specimen tested in air (see Figure 5). The morphology of the fracture surface is also changed; Figure 6 shows a reduction in ductile rupture (i.e., void growth and coalescence) in the specimen tested in hydrogen. This behavior is consistent with what has been observed in studies, previously.

Much of the data generated from study in this area has yielded data which is more applicable to academic study of the subject. In order to provide a consensus of properties for design of hydrogen systems, a mechanical properties database should be developed for each material selected for this type of application. This requires the use a statistically valid number of specimens for each testing condition. In the present study, 6 test conditions with 6 specimens per test condition were performed (36 specimens in all). The test matrix, shown in Table II, lists the sample names for each specimen in the test program. On occasion, difficulties with the data acquisition system or test equipment required an additional specimen be tested. To eliminate confusion additional specimen numbers were used. Hence, the test matrix shows (see Table II) shows specimen numbers high than 6, in some cases. The engineering stress vs. engineering strain curves are presented in Figures 7-42 for the specimens listed in Table II. The mechanical property data for the test matrix is presented in Table III. From the presentation of the data, it can be observed that no significant changes occur in yield stress or ultimate tensile stress. However, a mild reduction in elongation to failure is observed. Reduction of area is a property that must be measured after the test and is very sensitive to small differences in the deformation behavior of the material. Although the reduction of area data was not available for this report, Figures 5 and 6 indicate that the reduction of area property is expected to be consistent with the elongation to failure exhibited in Table III.

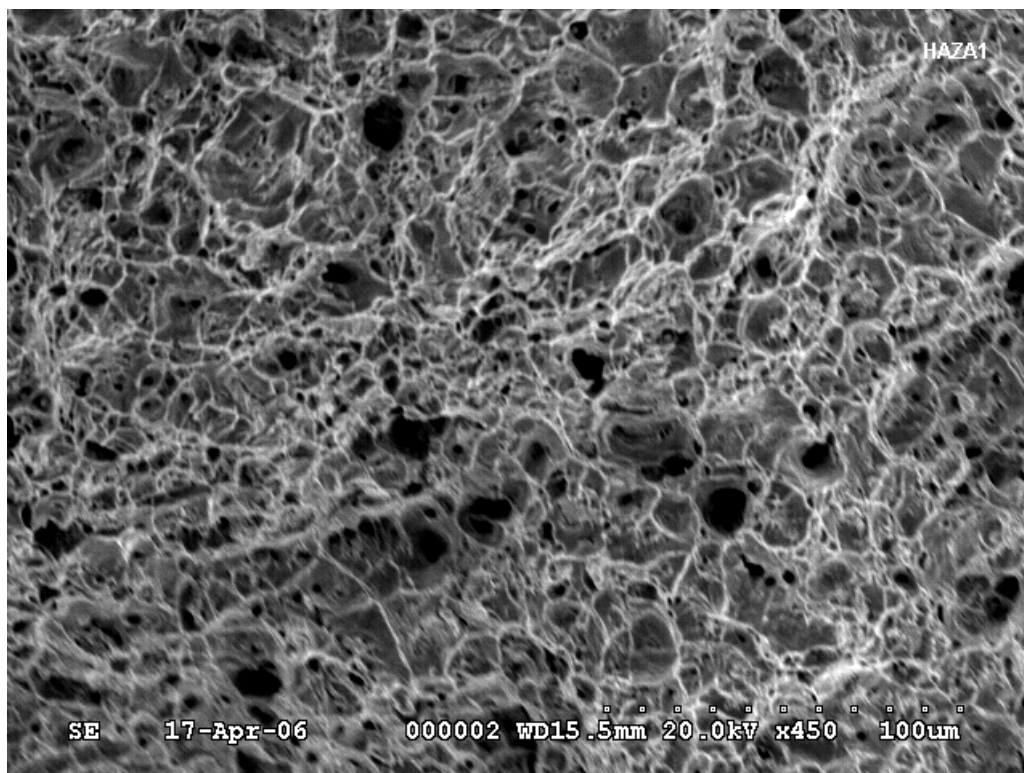


(a)

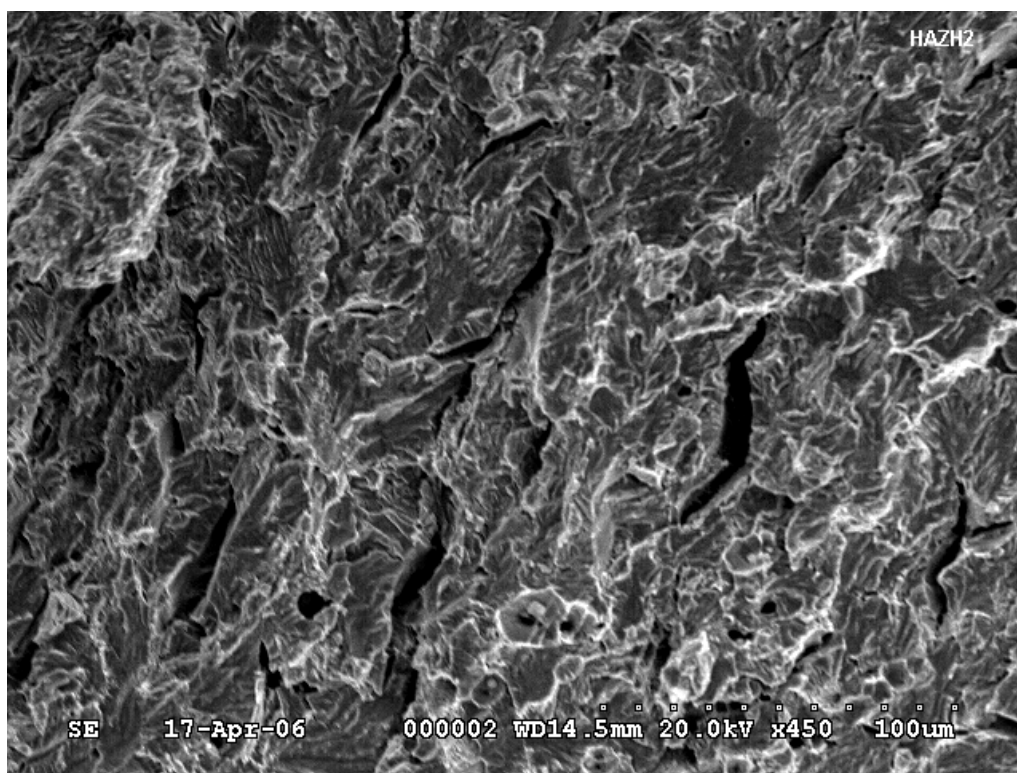


(b)

Figure 5: Low Magnification Scanning Electron Micrographs of the Fracture Surface from HAZ Specimens Tested in Air (a) and 1500 psig Hydrogen (b)



(a)



(b)

Figure 6 Higher Magnification Scanning Electron Micrographs of the Fracture Surface from HAZ Specimens Tested in Air (a) and 1500 psig Hydrogen (b)

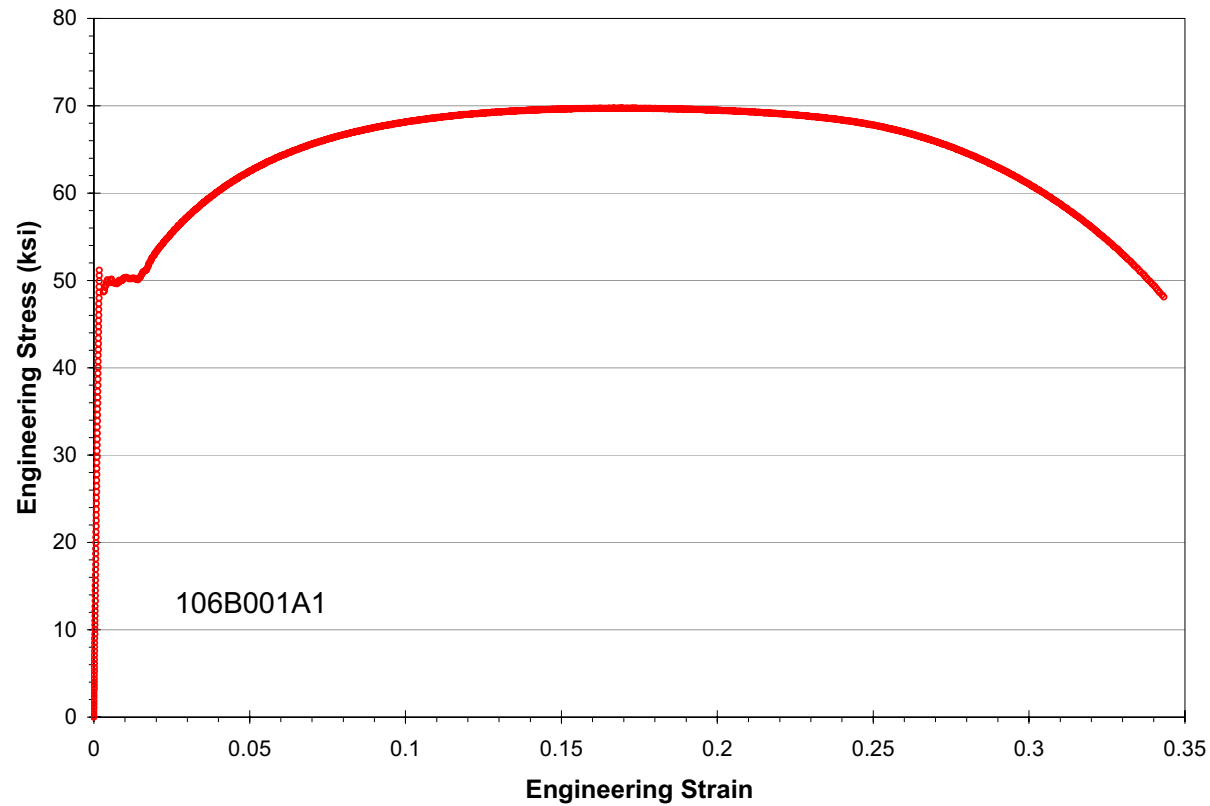


Figure 7: Engineering Stress vs. Strain for Sample # 106B001A1 (Base Metal in Air)

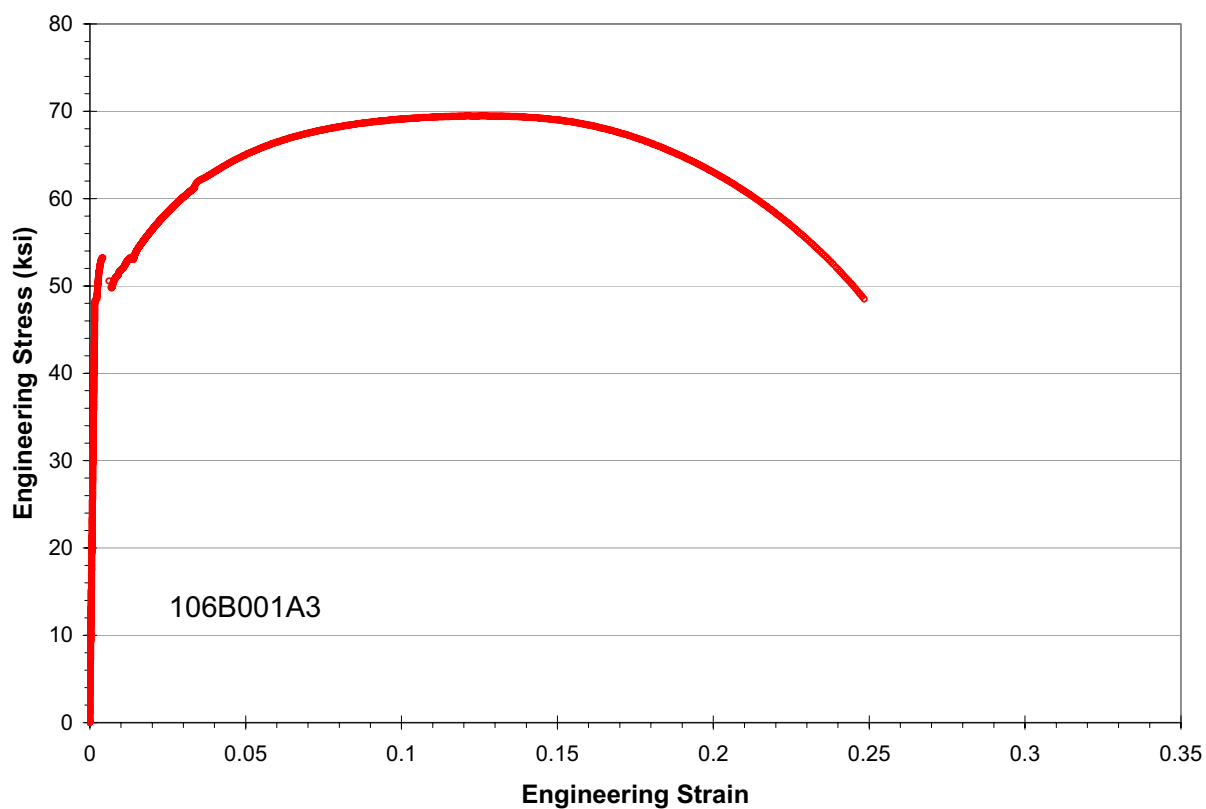


Figure 8: Engineering Stress vs. Strain for Sample # 106B001A3 (Base Metal in Air)

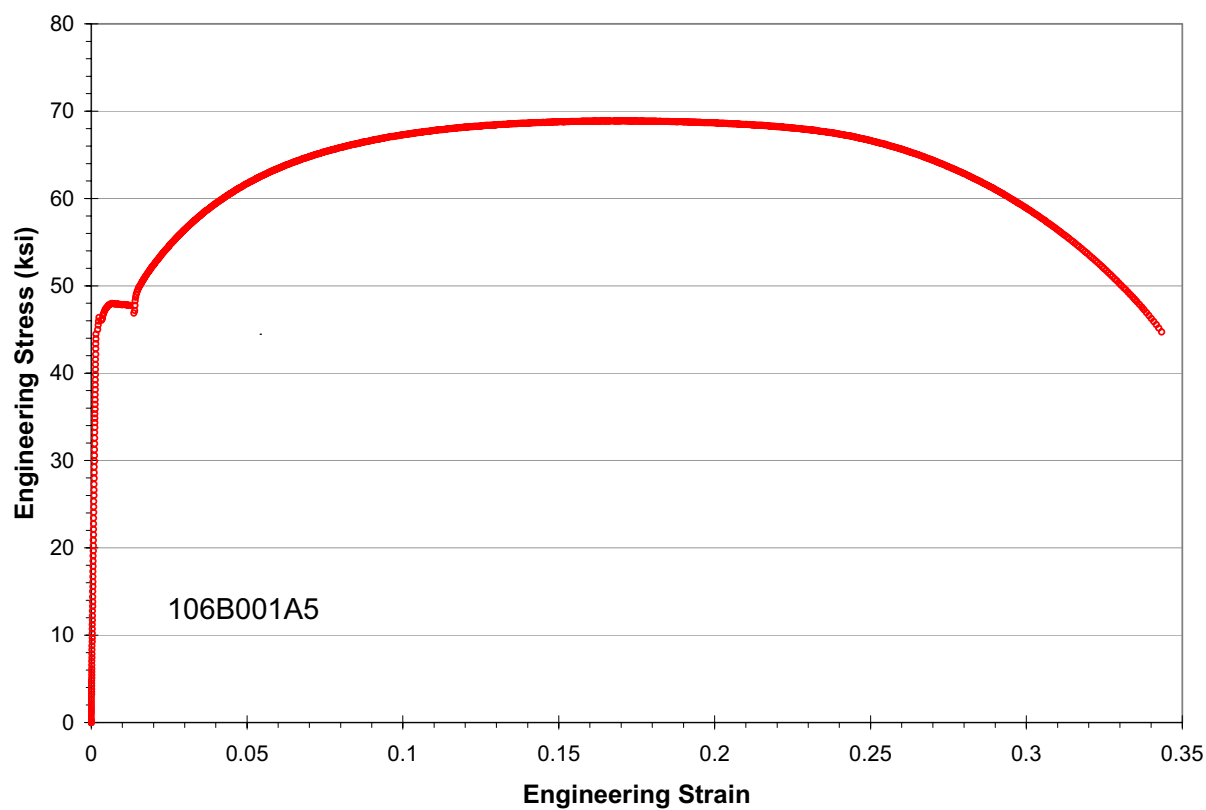


Figure 9: Engineering Stress vs. Strain for Sample # 106B001A5 (Base Metal in Air)

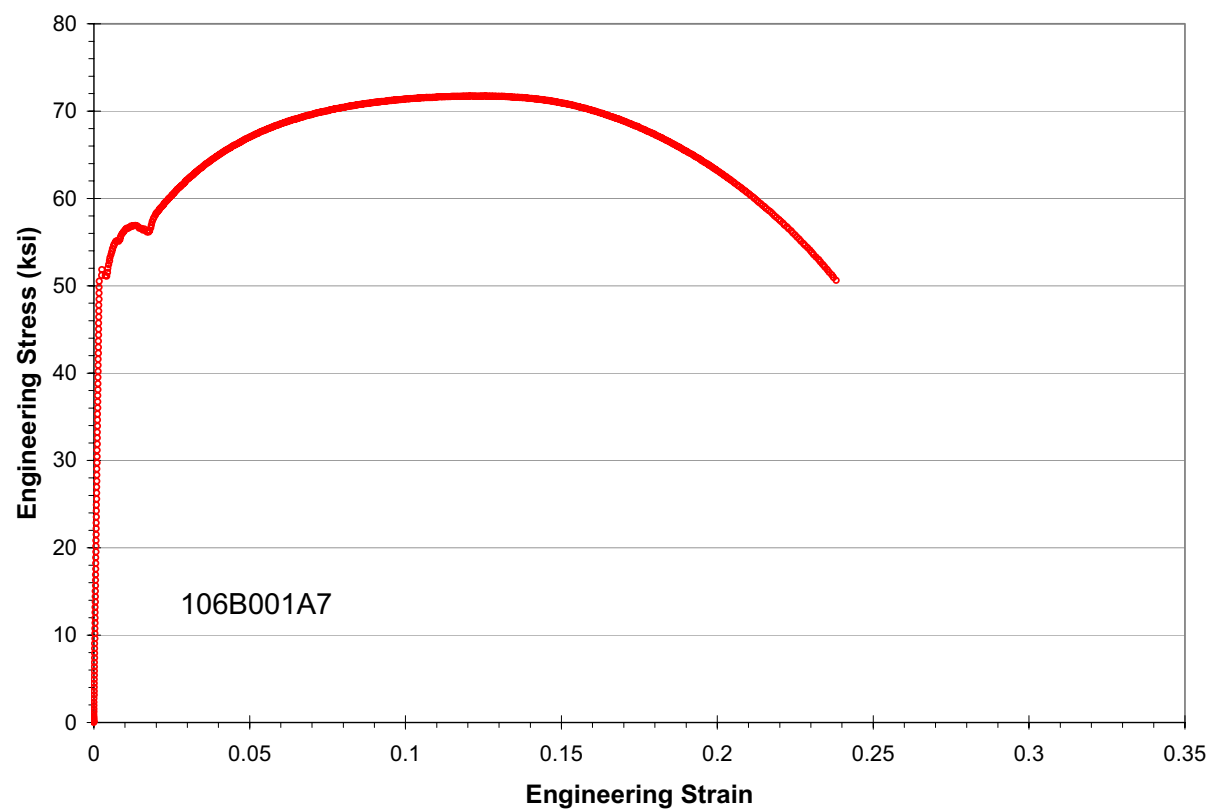


Figure 10: Engineering Stress vs. Strain Sample # 106B001A7 (Base Metal in Air)

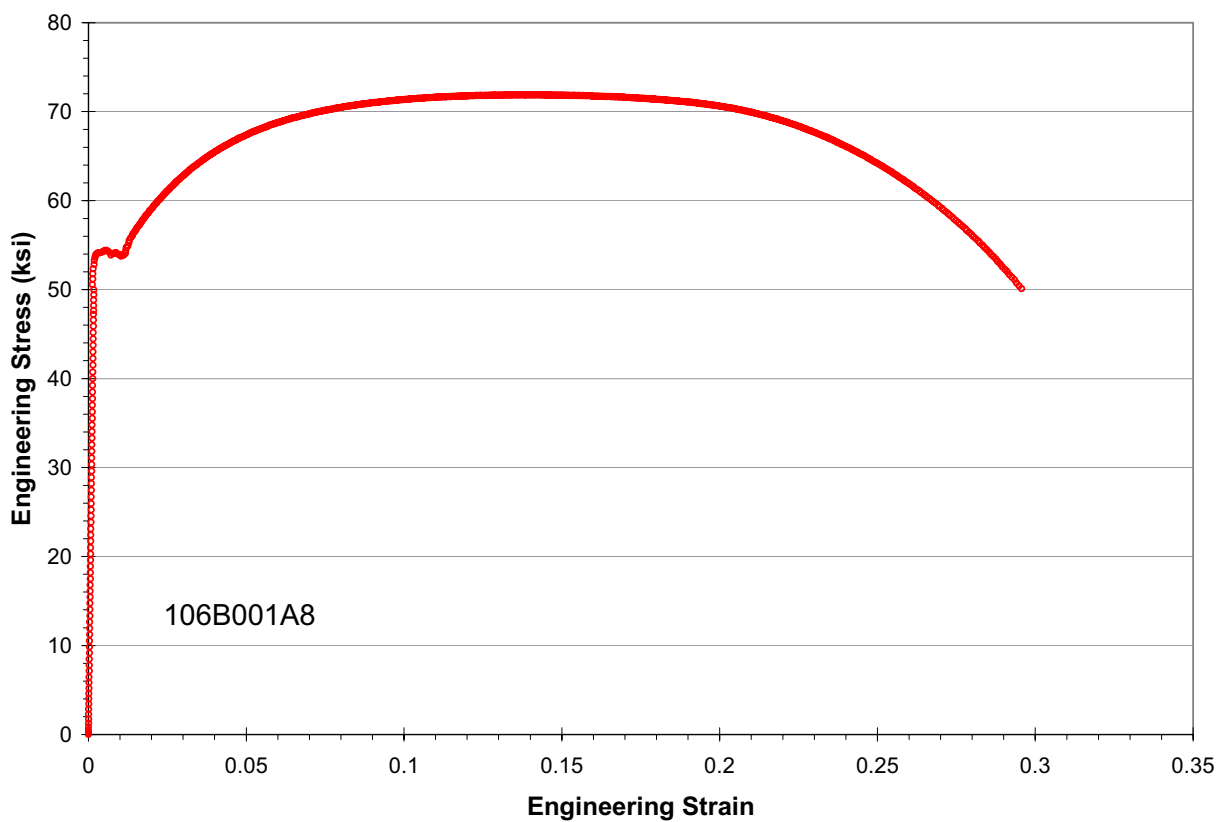


Figure 11: Engineering Stress vs. Strain for Sample # 106B001A8 (Base Metal in Air)

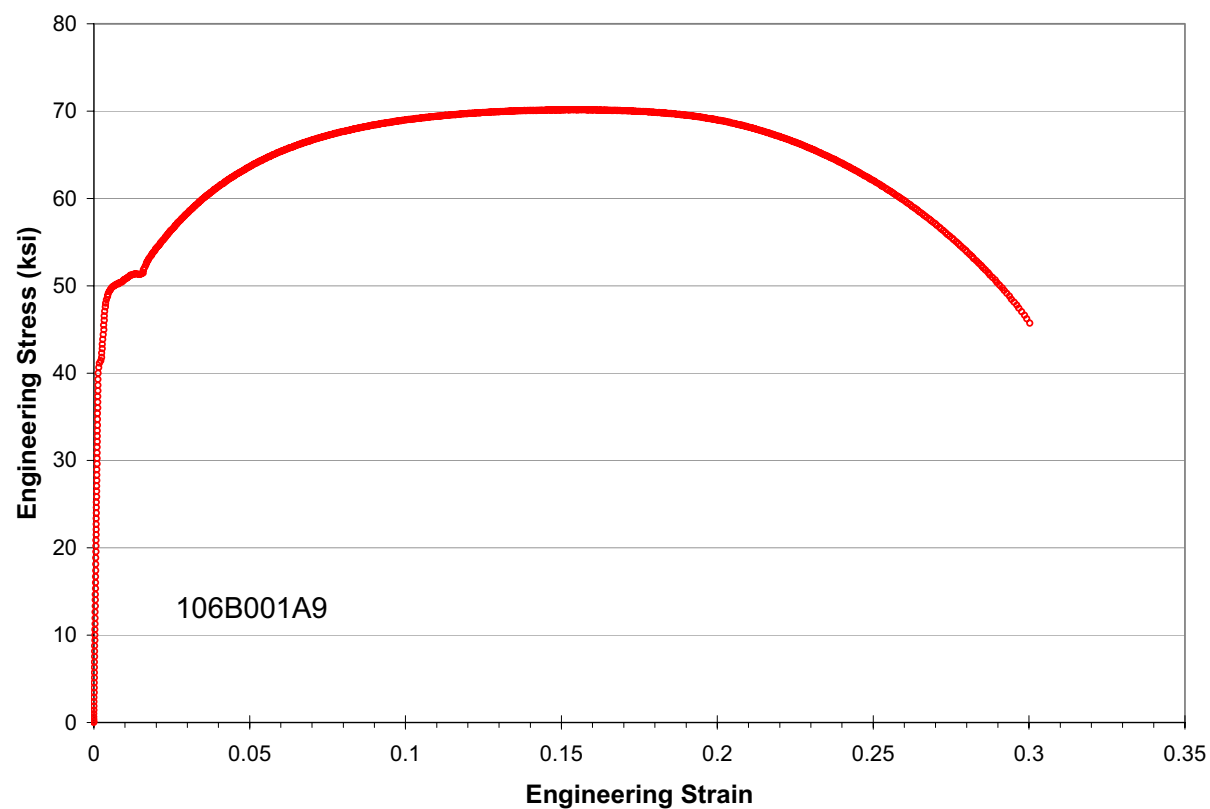


Figure 12: Engineering Stress vs. Strain for Sample # 106B001A9 (Base Metal in Air)

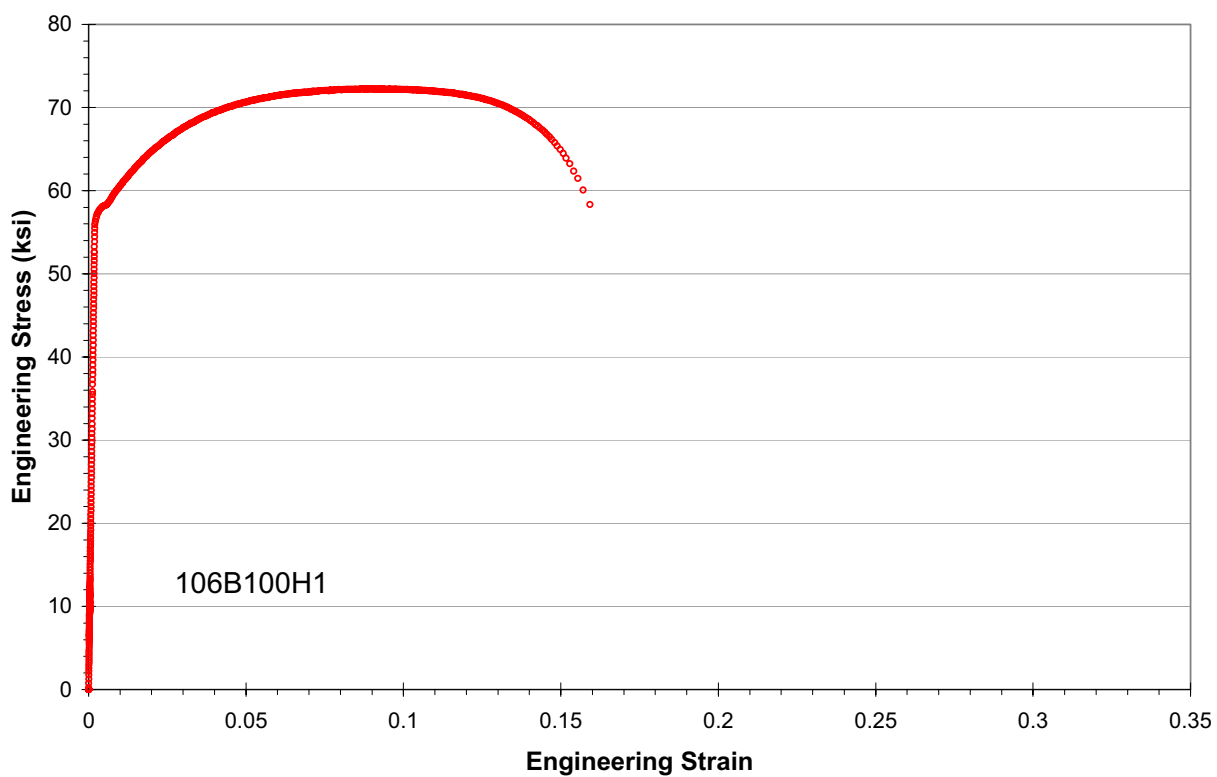


Figure 13: Engineering Stress vs. Strain for Sample # 106B100H1 (Base Metal in 1500 psig H₂)

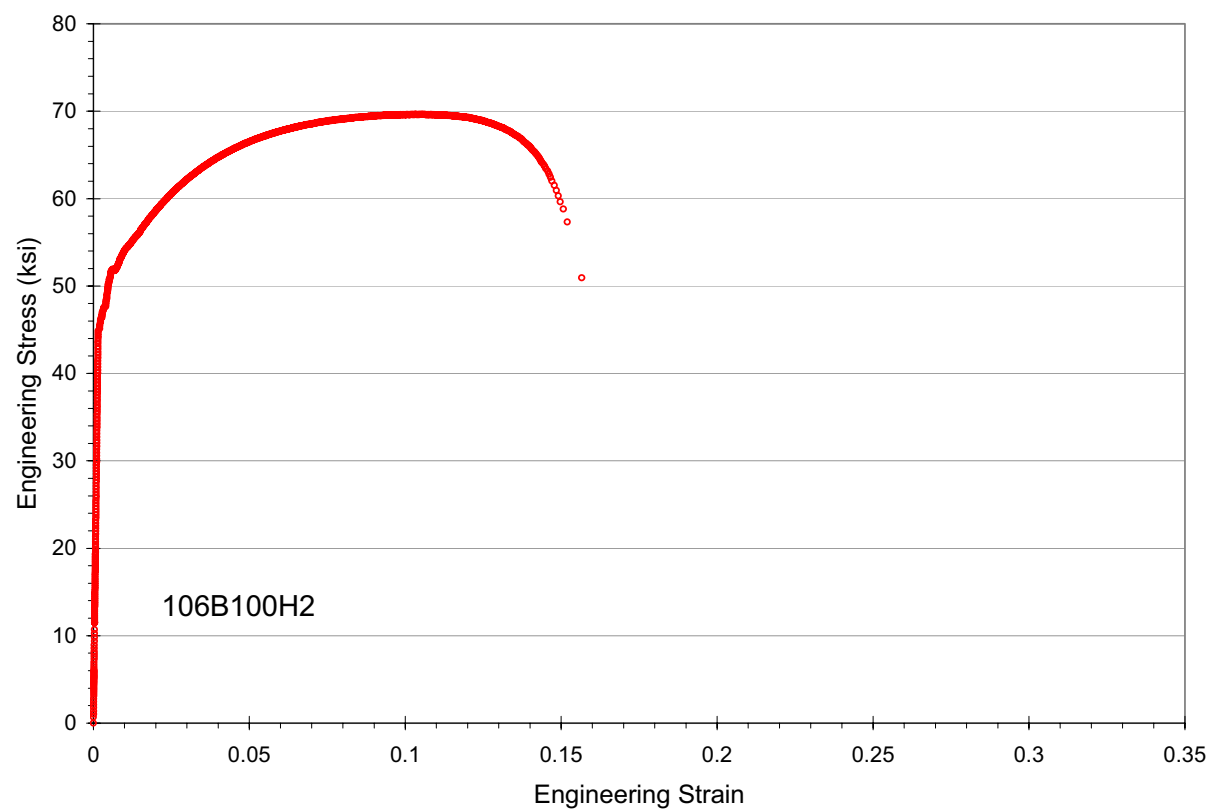


Figure 14: Engineering Stress vs. Strain for Sample # 106B100H2 (Base Metal in 1500 psig H₂)

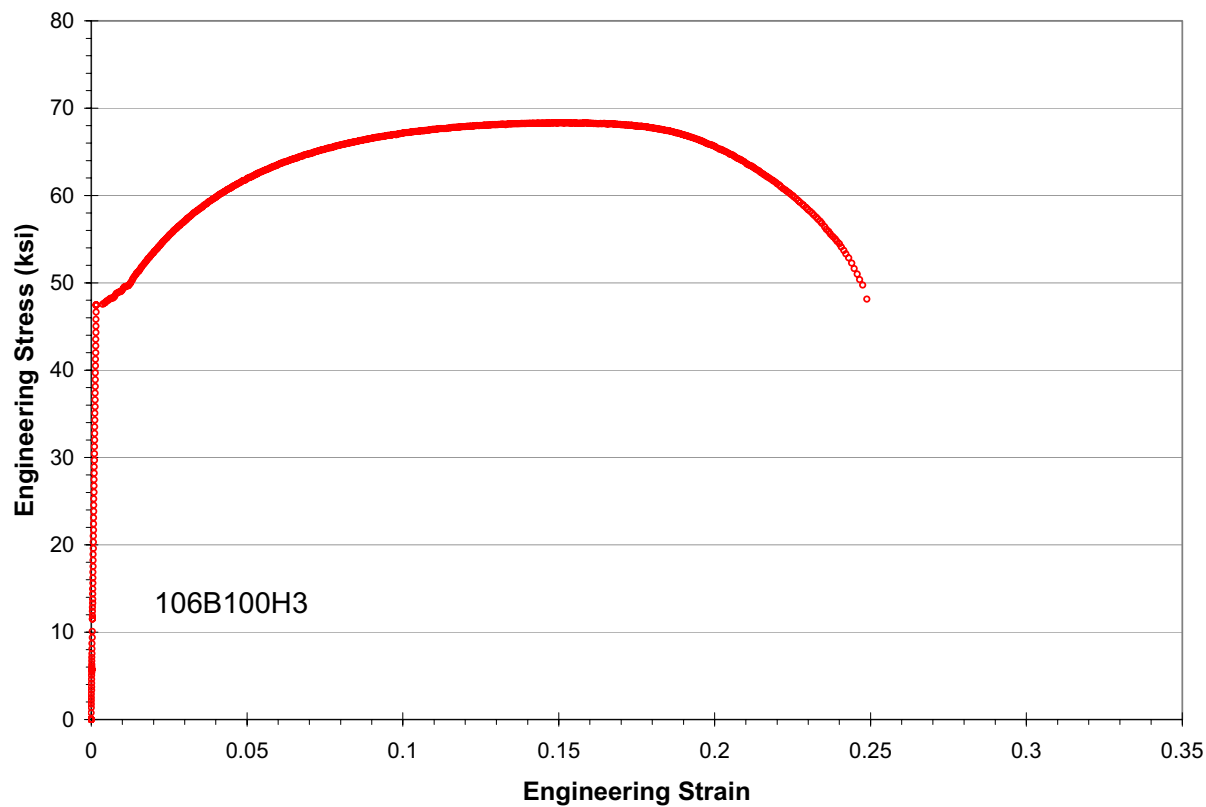


Figure 15: Engineering Stress vs. Strain for Sample # 106B100H3 (Base Metal in 1500 psig H₂)

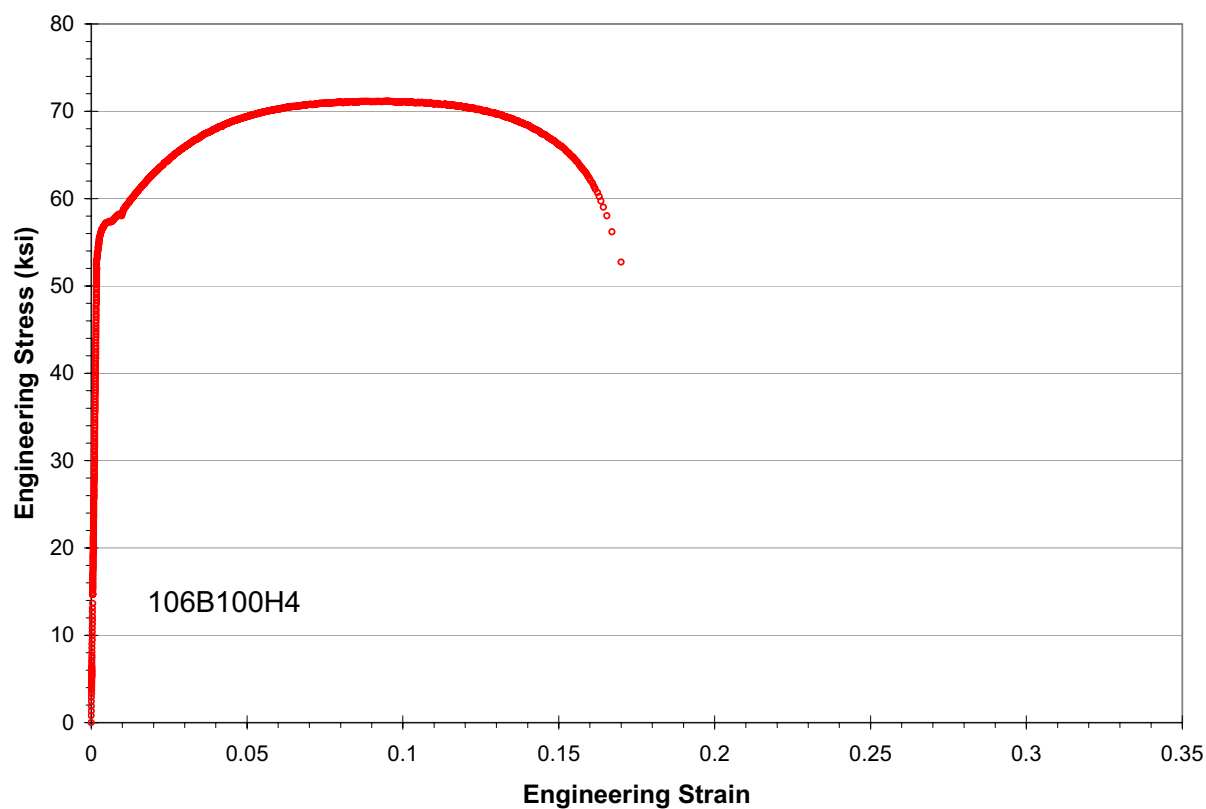


Figure 16: Engineering Stress vs. Strain for Sample # 106B100H4 (Base Metal in 1500 psig H₂)

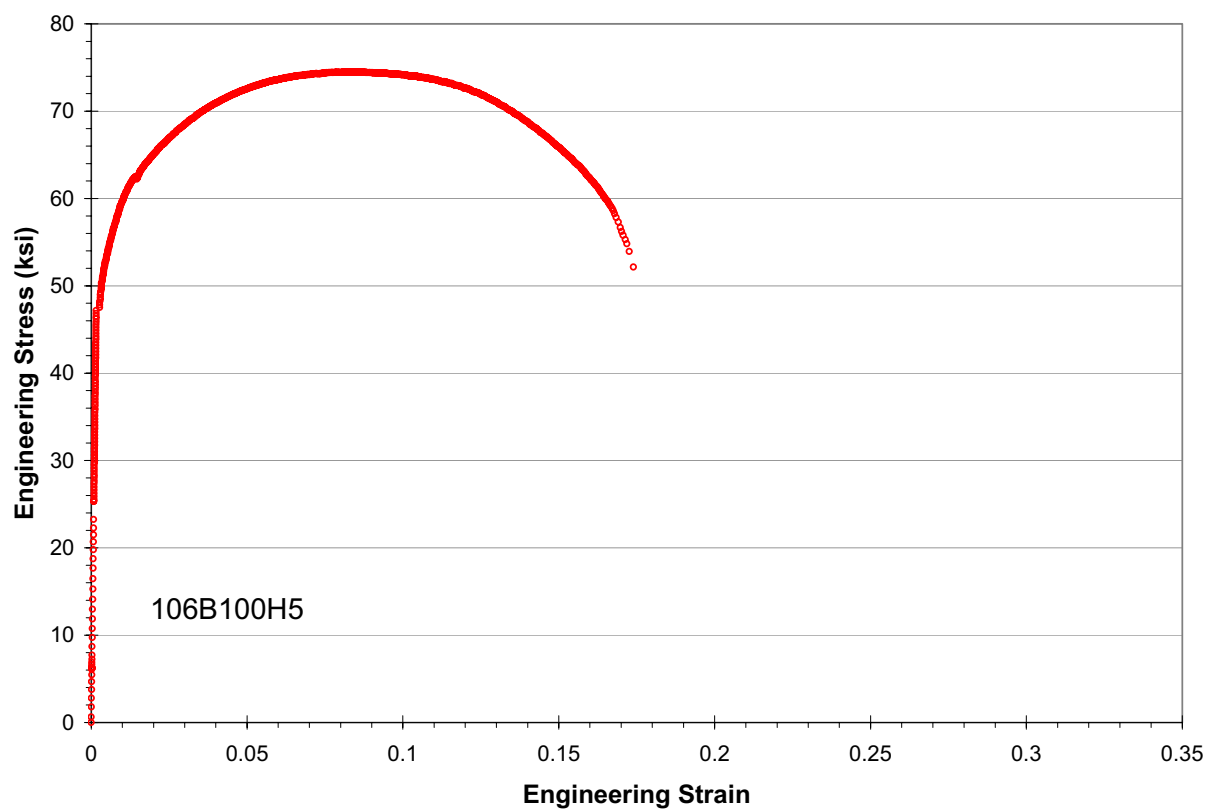


Figure 17: Engineering Stress vs. Strain for Sample # 106B100H5 (Base Metal in 1500 psig H₂)

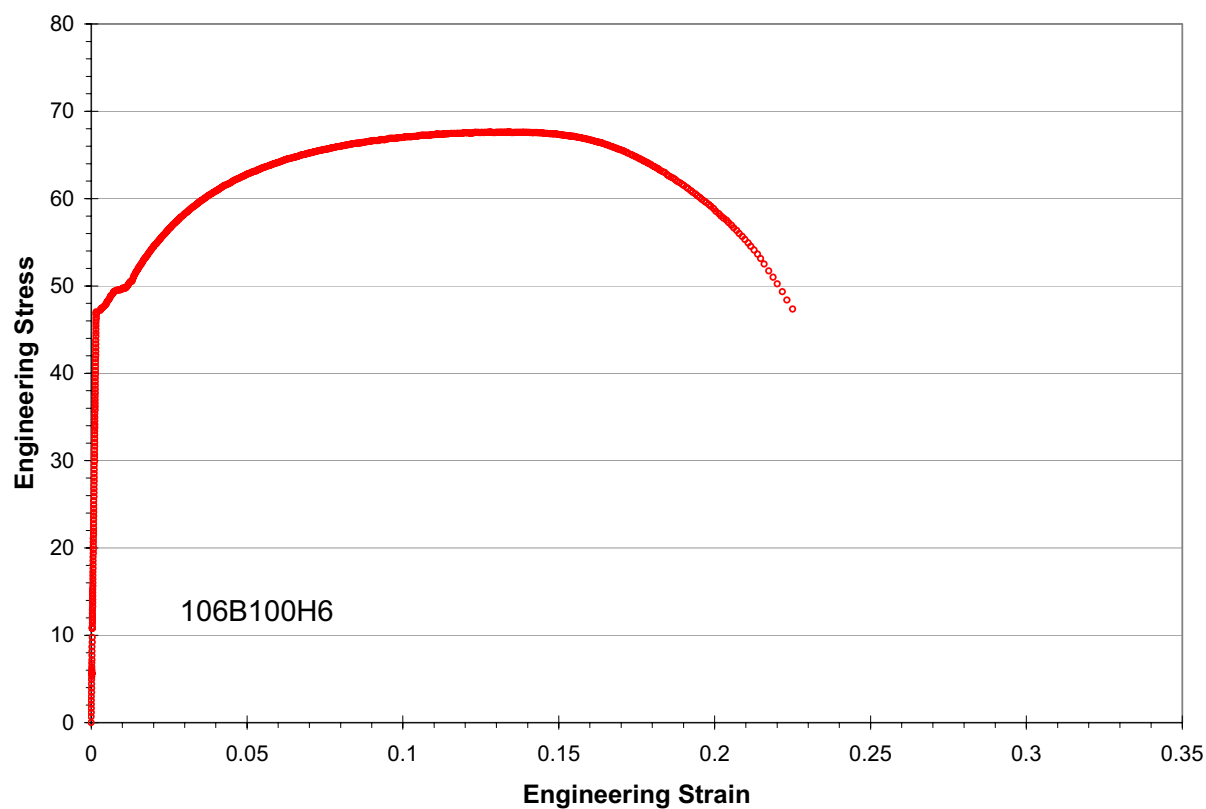


Figure 18: Engineering Stress vs. Strain for Sample # 106B100H6 (Base Metal in 1500 psig H₂)

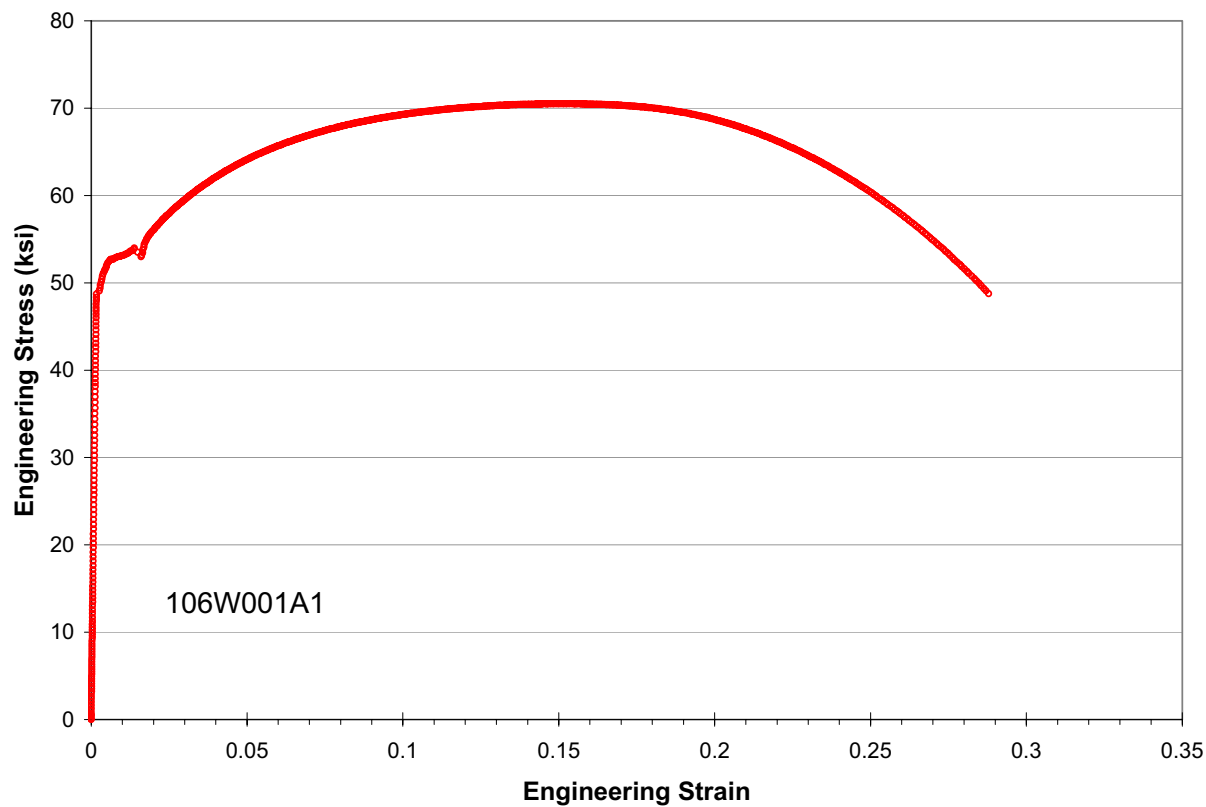


Figure 19: Engineering Stress vs. Strain for Sample # 106W001A1 (Weld Metal in Air)

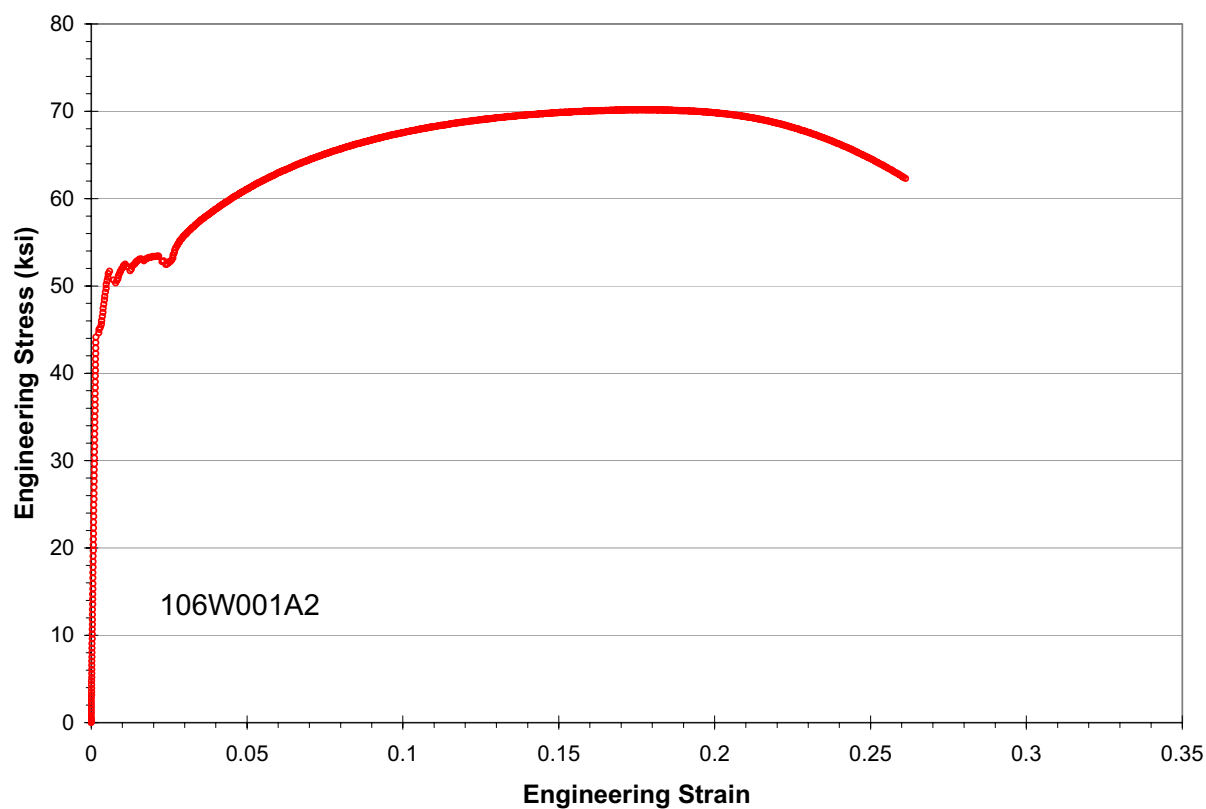


Figure 20: Engineering Stress vs. Strain for Sample # 106W001A2 (Weld Metal in Air)

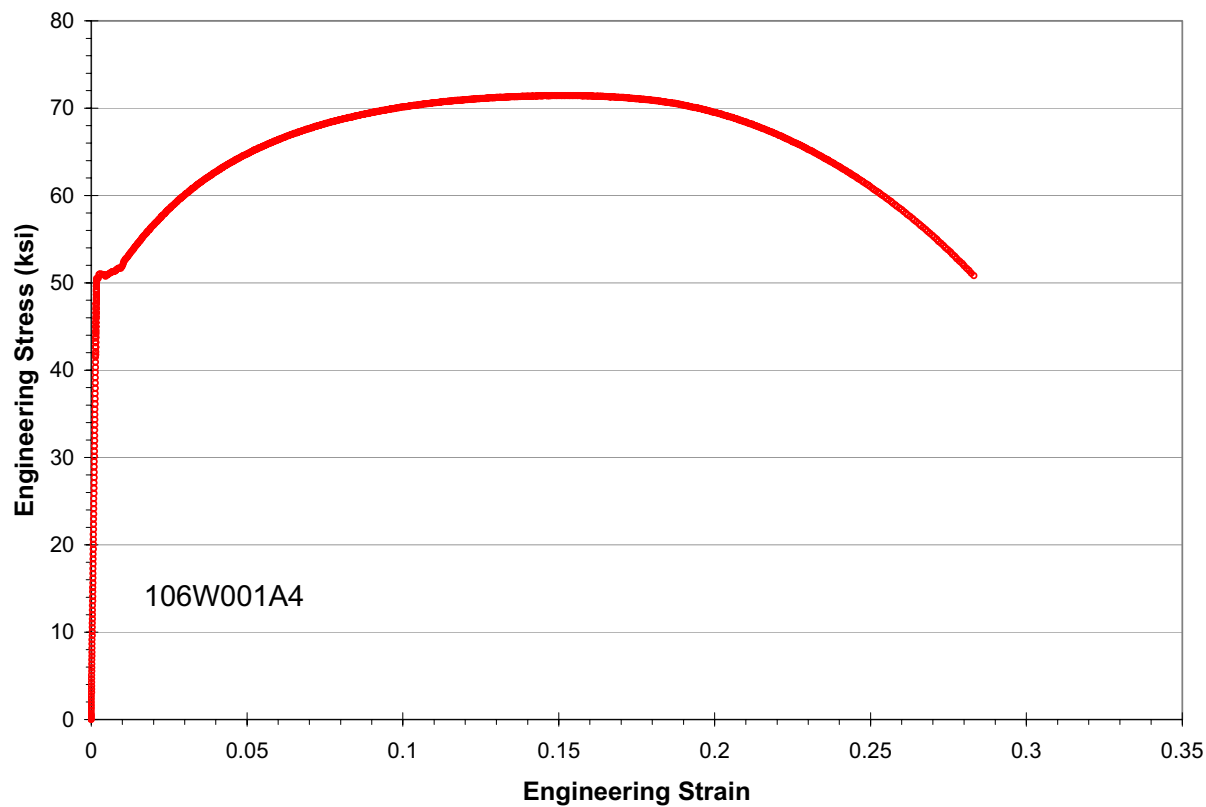


Figure 21: Engineering Stress vs. Strain for Sample # 106W001A4 (Weld Metal in Air)

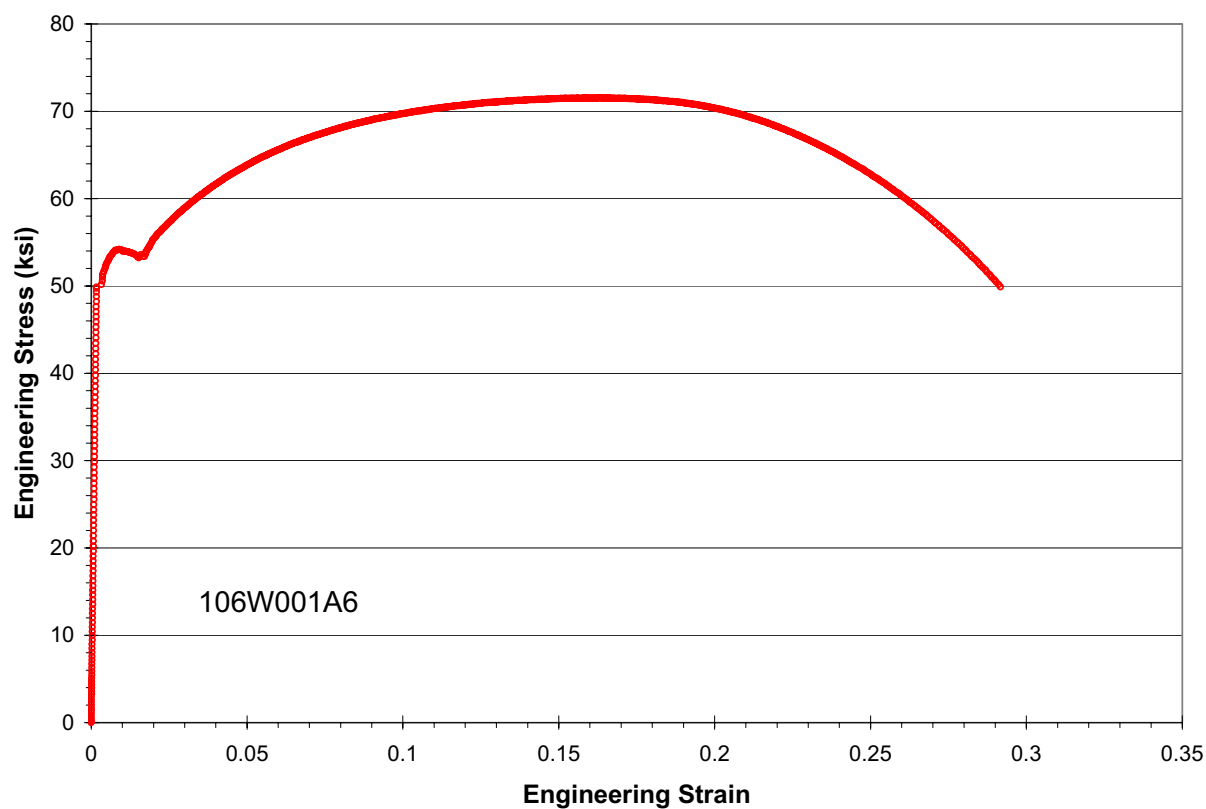


Figure 22: Engineering Stress vs. Strain for Sample # 106W001A6 (Weld Metal in Air)

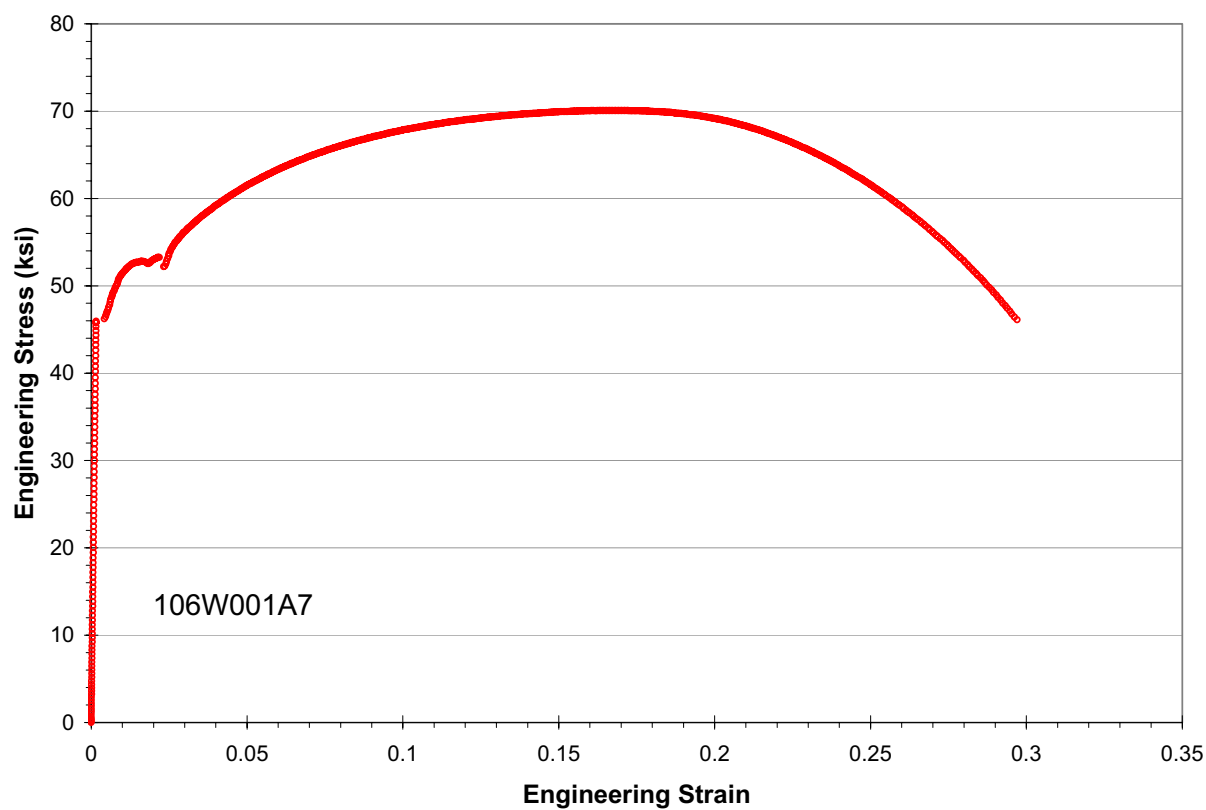


Figure 23: Engineering Stress vs. Strain for Sample # 106W001A7 (Weld Metal in Air)

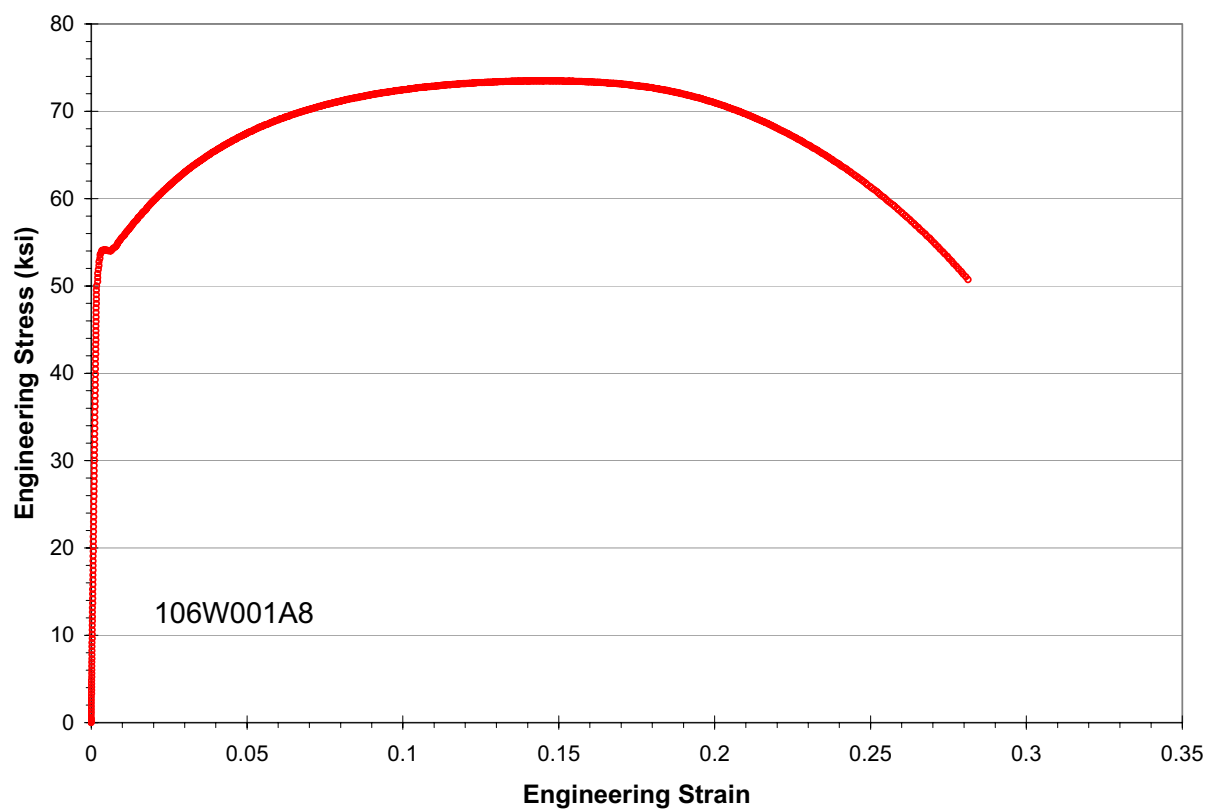


Figure 24: Engineering Stress vs. Strain for Sample # 106W001A8 (Weld Metal in Air)

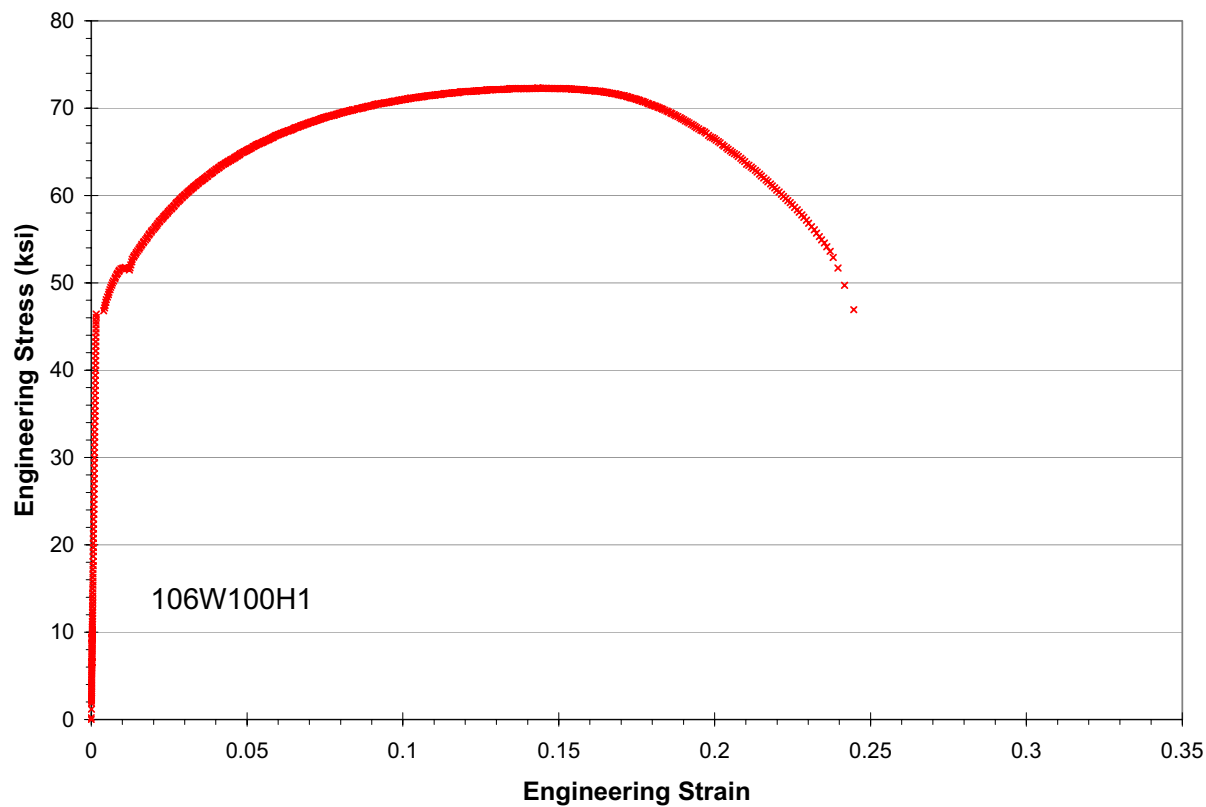


Figure 25: Engineering Stress vs. Strain for Sample # 106W100H1 (Weld Metal in 1500 psig H₂)

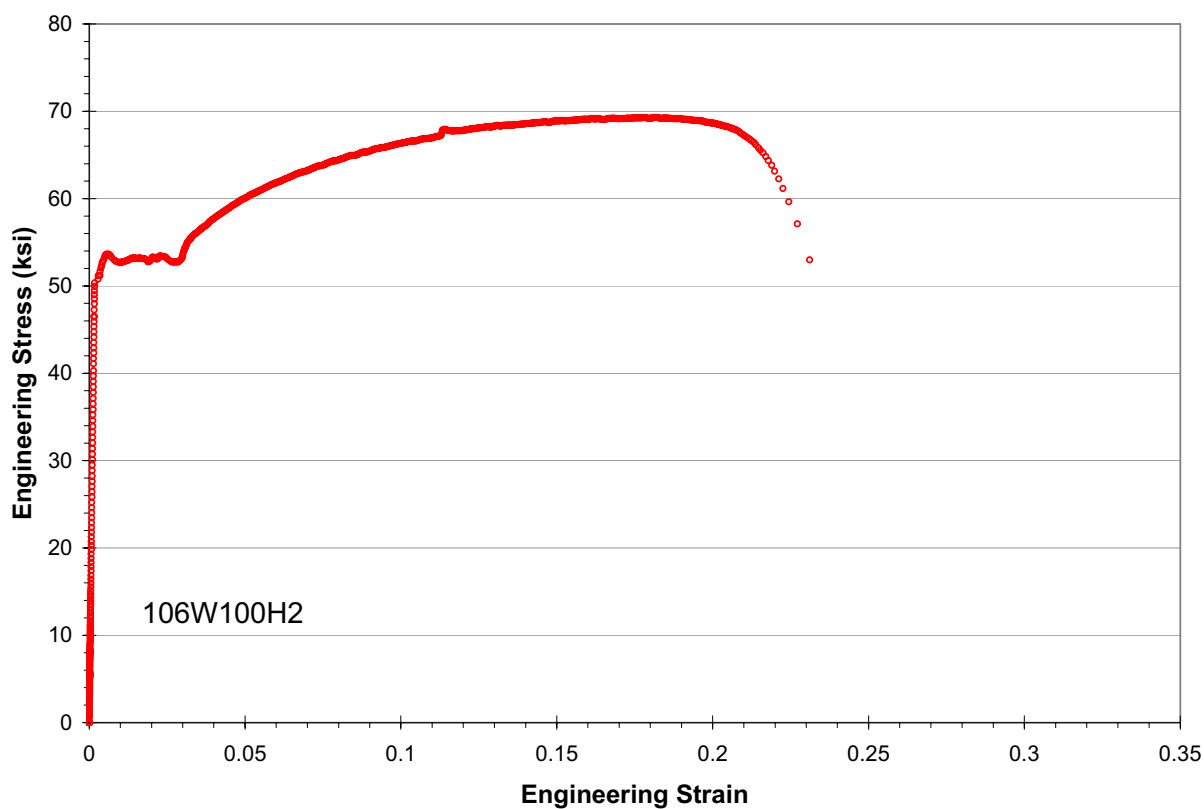


Figure 26: Engineering Stress vs. Strain for Sample # 106W100H2 (Weld Metal in 1500 psig H₂)

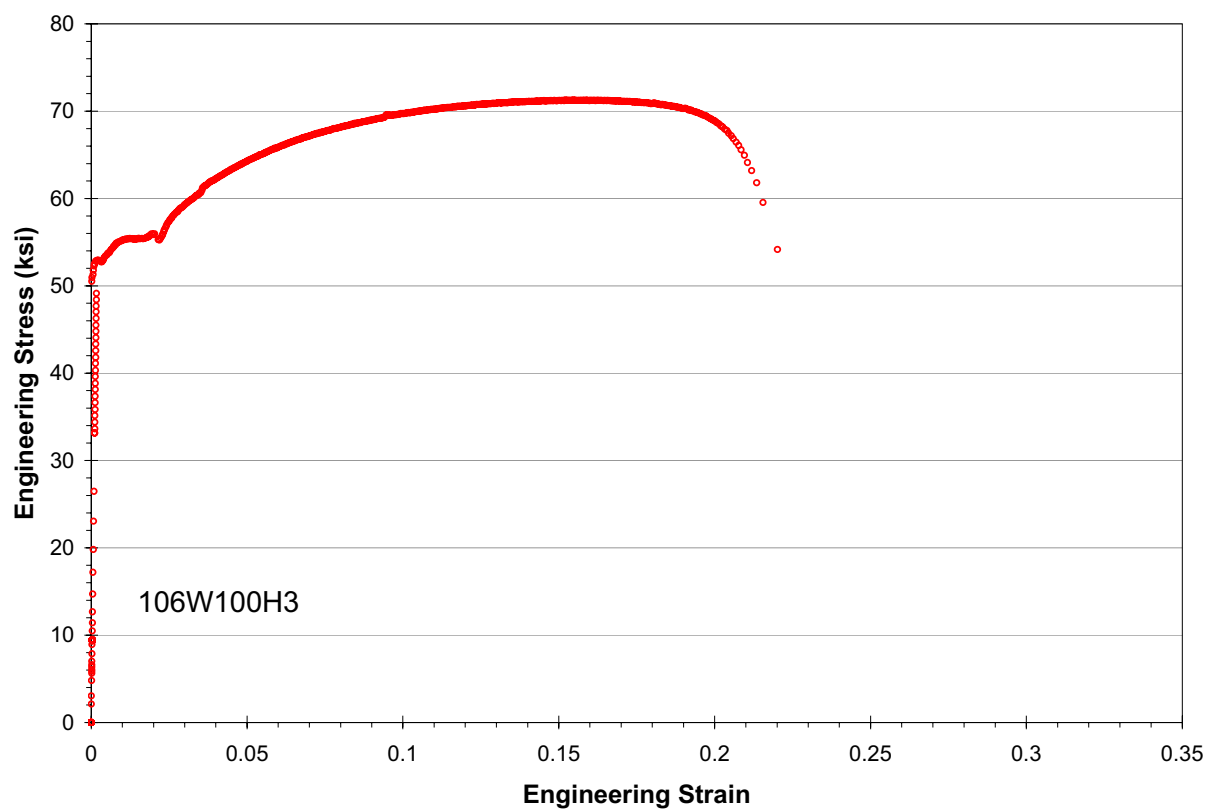


Figure 27: Engineering Stress vs. Strain for Sample # 106W100H3 (Weld Metal in 1500 psig H₂)

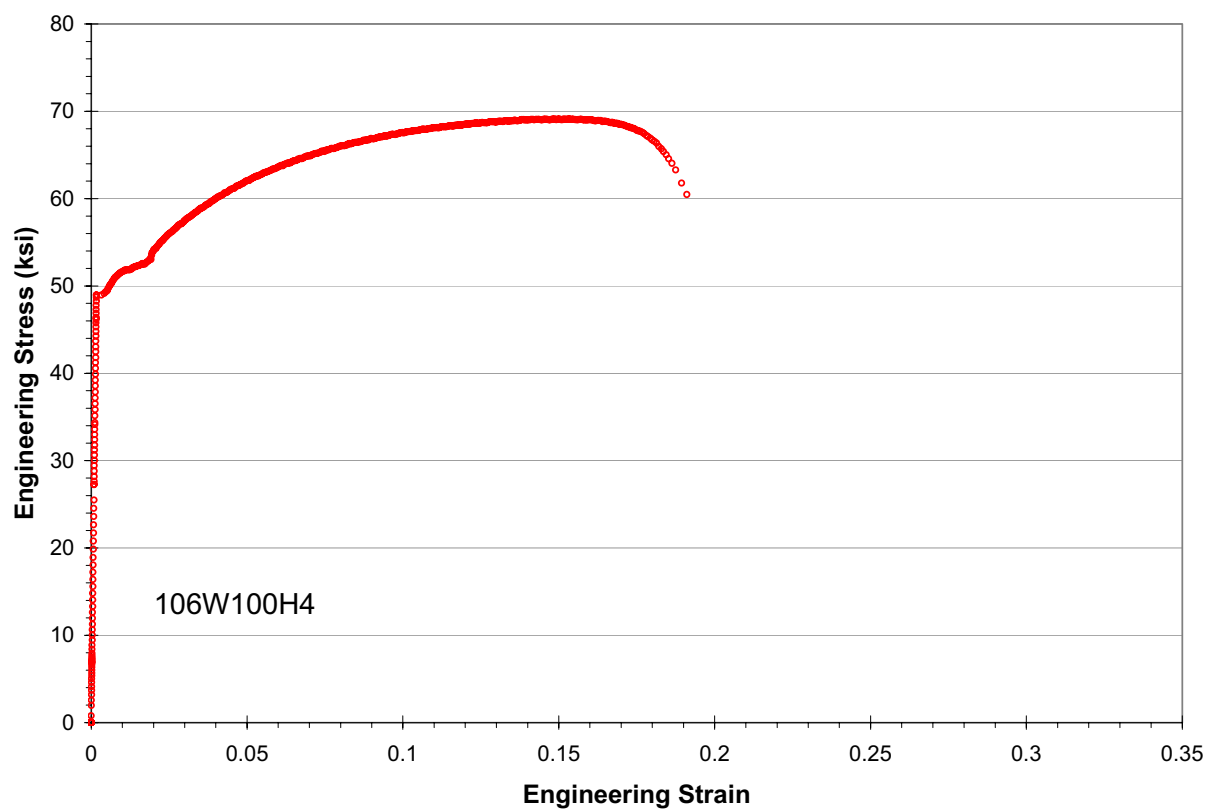


Figure 28: Engineering Stress vs. Strain for Sample # 106W100H4 (Weld Metal in 1500 psig H₂)

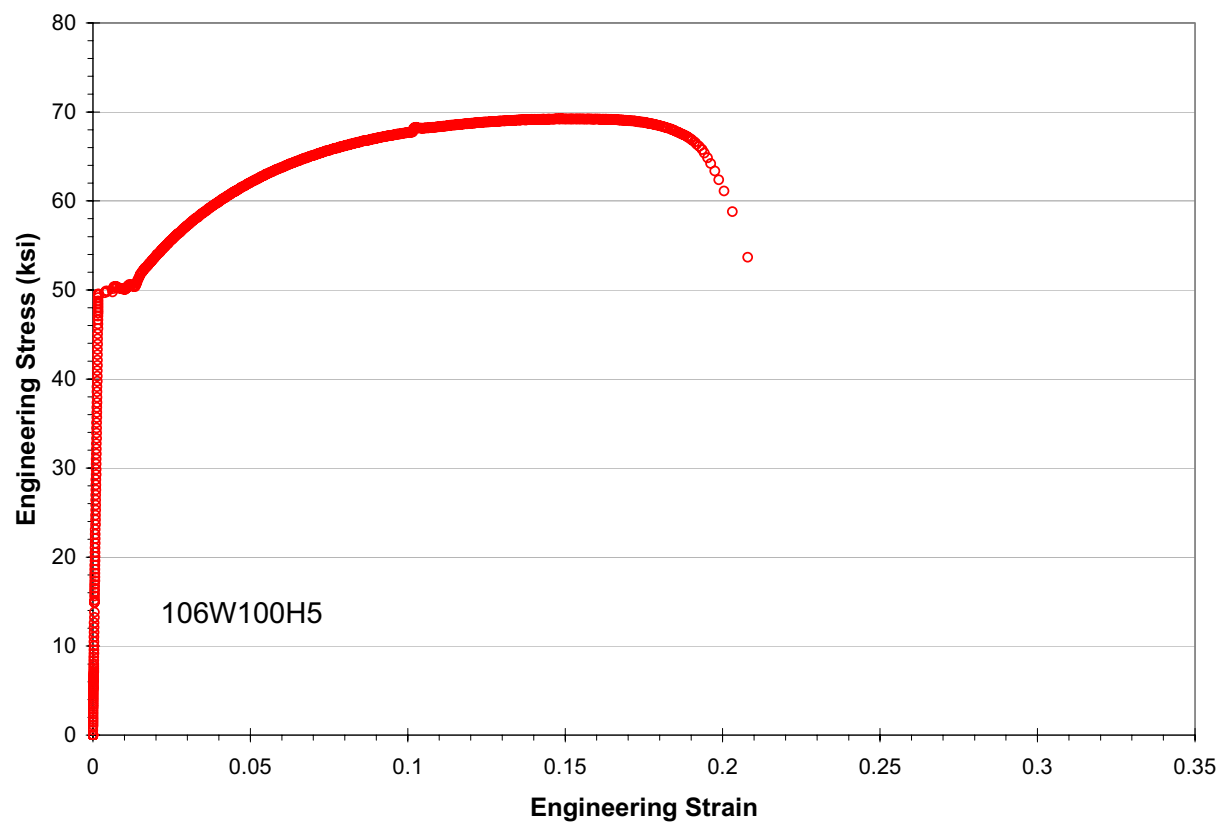


Figure 29: Engineering Stress vs. Strain for Sample # 106W100H5 (Weld Metal in 1500 psig H₂)

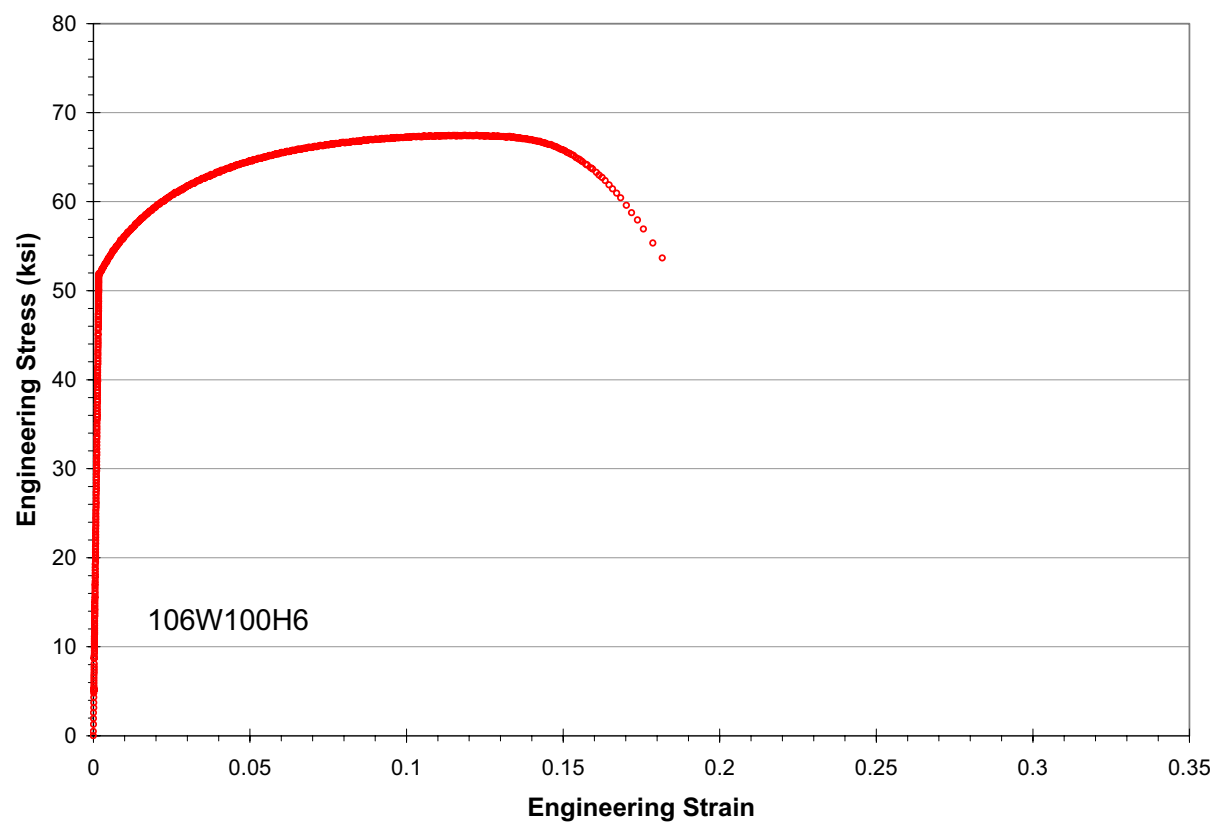


Figure 30: Engineering Stress vs. Strain for Sample # 106W100H6 (Weld Metal in 1500 psig H₂)

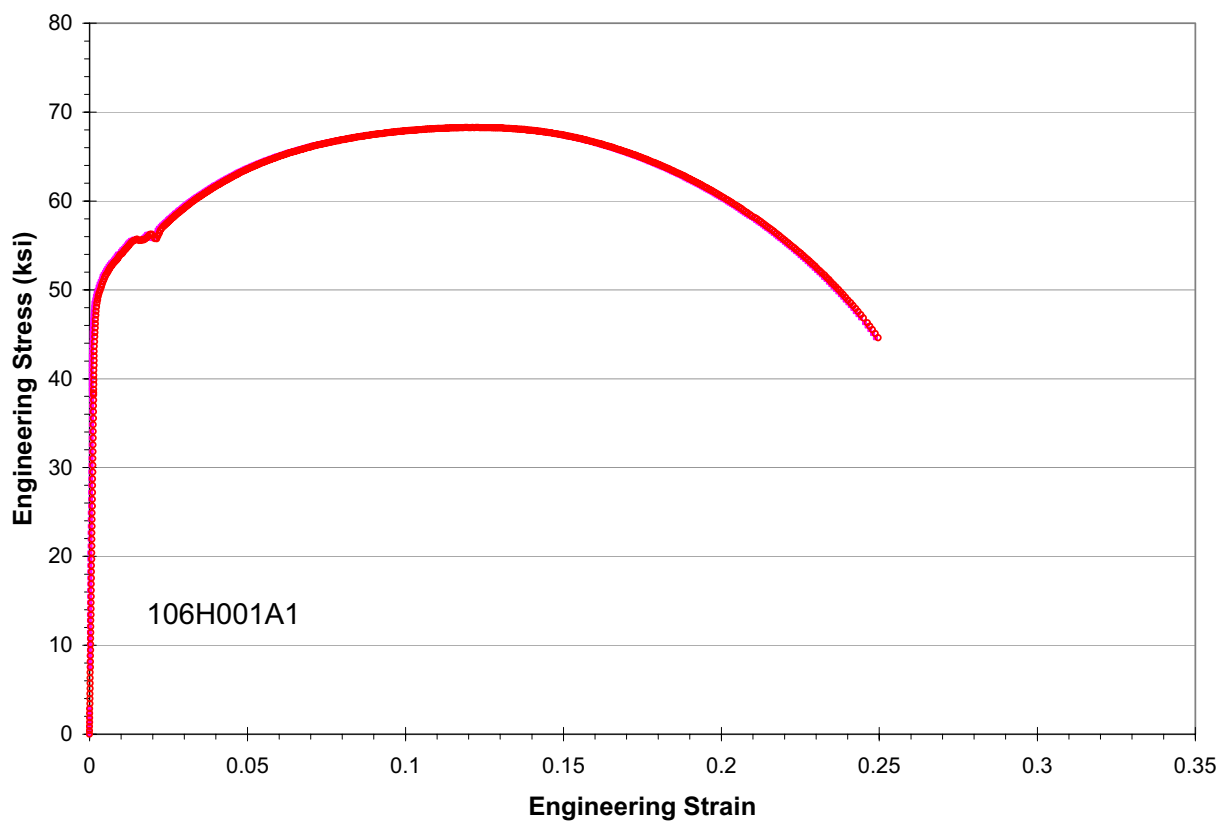


Figure 31: Engineering Stress vs. Strain for Sample # 106H001A1 (HAZ in Air)

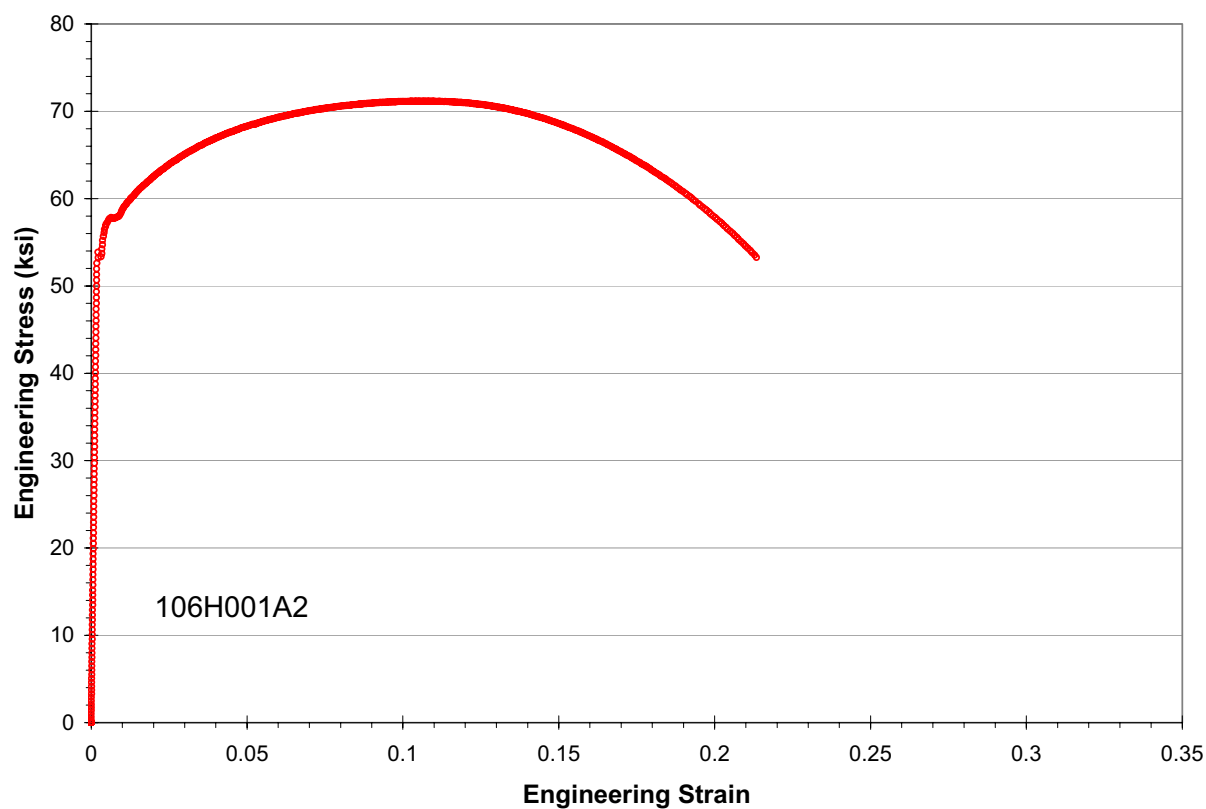


Figure 32: Engineering Stress vs. Strain for Sample # 106H001A2 (HAZ in Air)

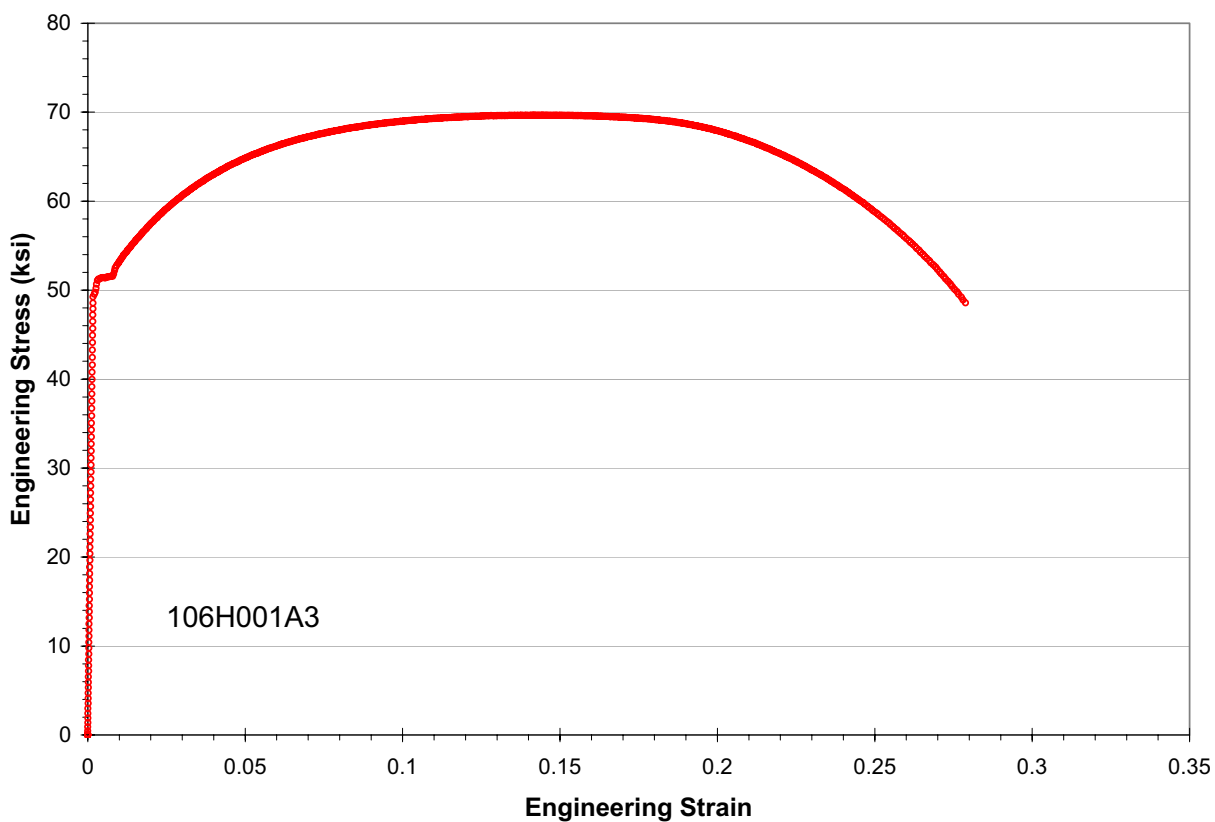


Figure 33: Engineering Stress vs. Strain for Sample # 106H001A3 (HAZ in Air)

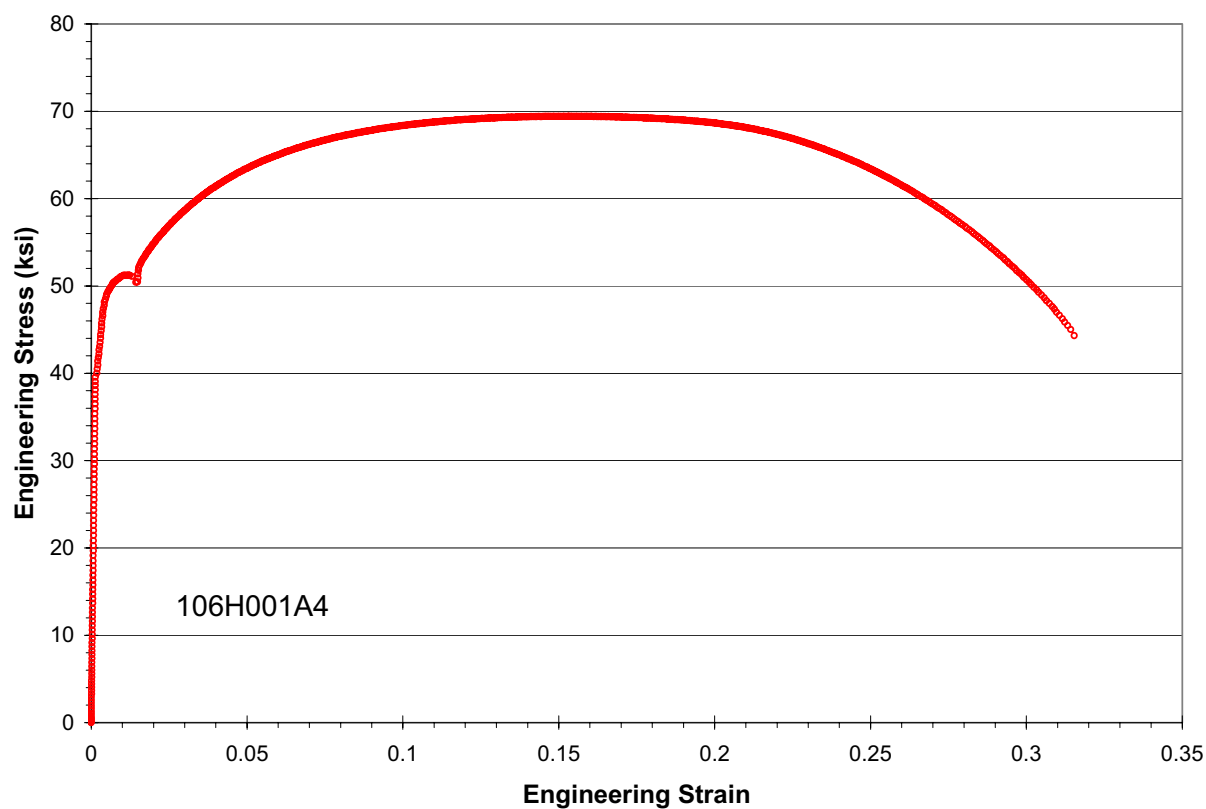


Figure 34: Engineering Stress vs. Strain for Sample # 106H001A4 (HAZ in Air)

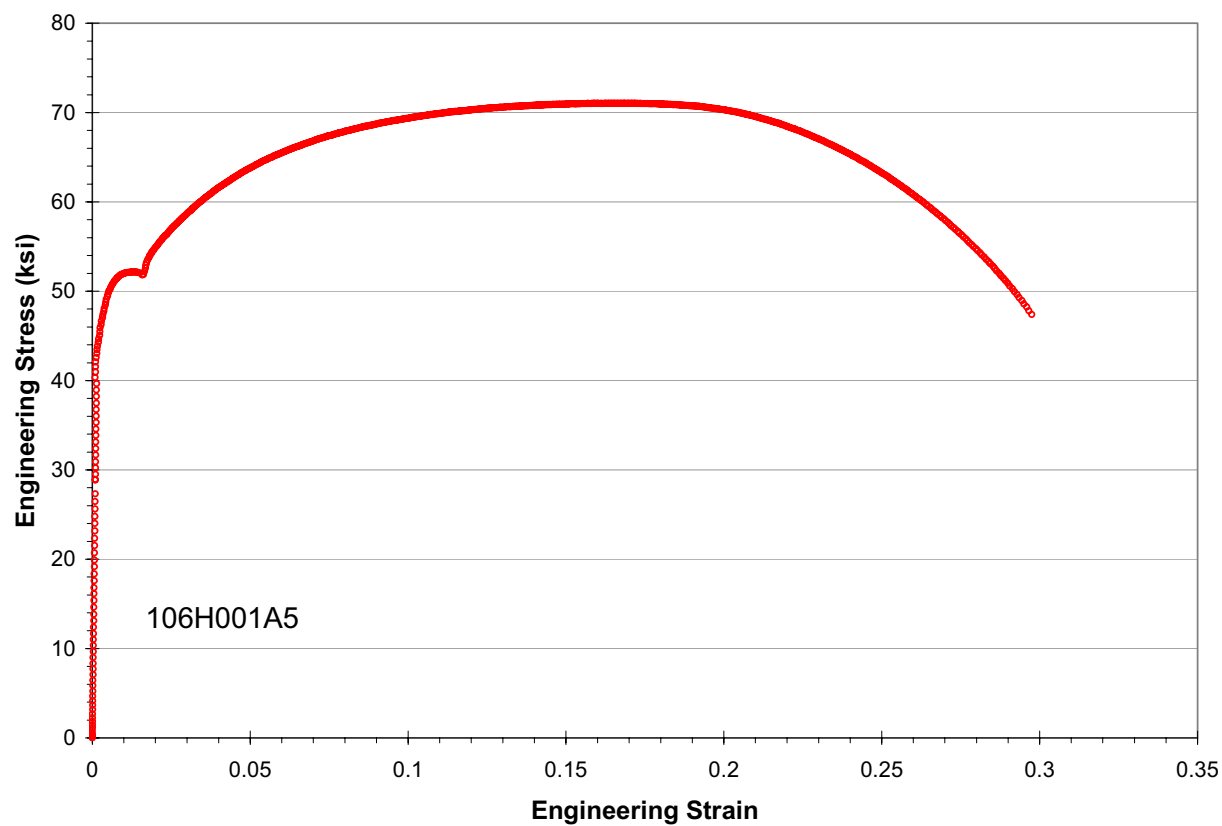


Figure 35: Engineering Stress vs. Strain for Sample # 106H001A5 (HAZ in Air)

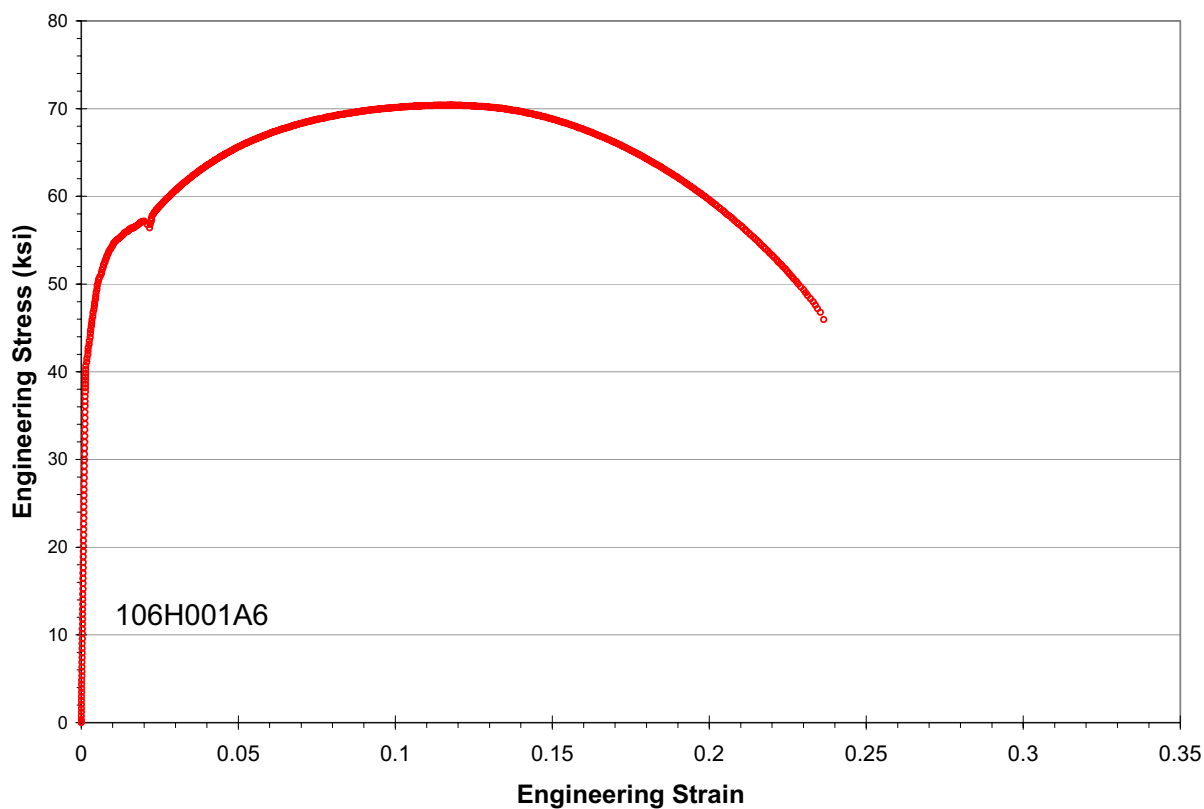


Figure 36: Engineering Stress vs. Strain for Sample # 106H001A6 (HAZ in Air)

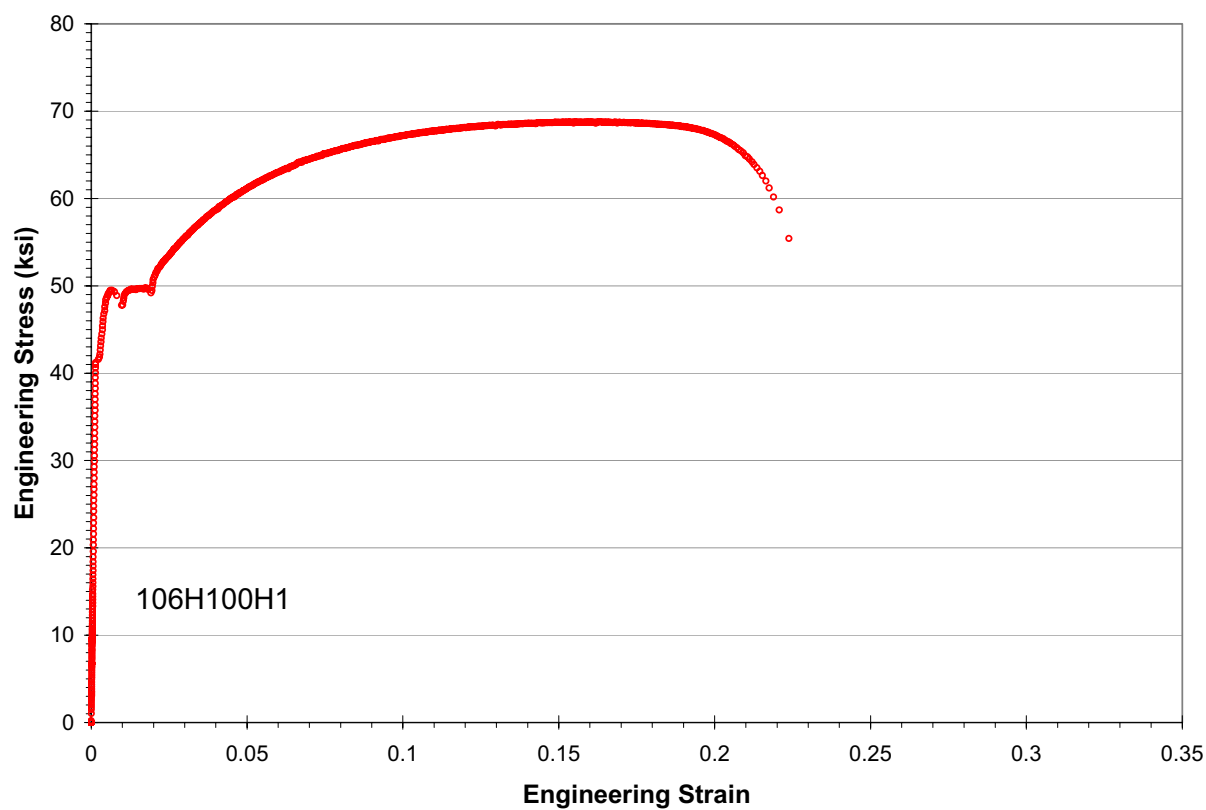


Figure 37: Engineering Stress vs. Strain for Sample # 106H100H1 (HAZ in 1500 psig H₂)

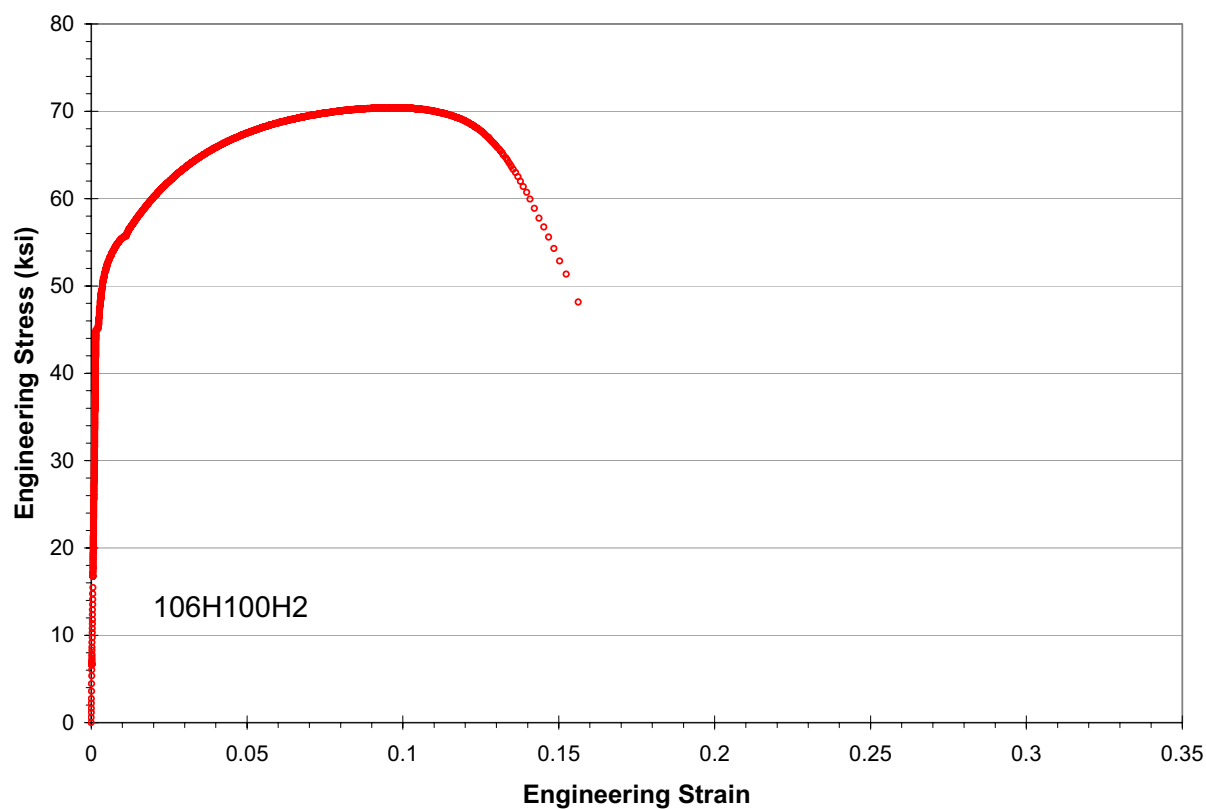


Figure 38: Engineering Stress vs. Strain for Sample # 106H100H1 (HAZ in 1500 psig H₂)

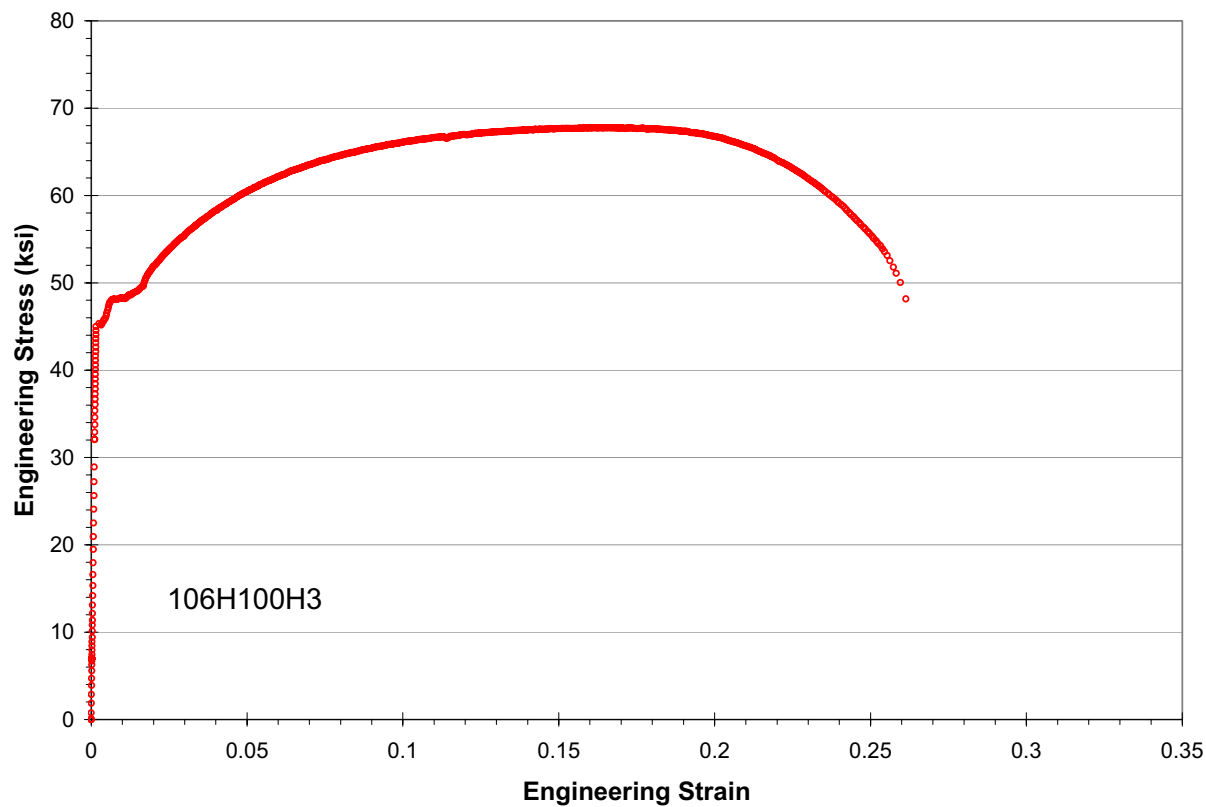


Figure 39: Engineering Stress vs. Strain for Sample # 106H100H1 (HAZ in 1500 psig H₂)

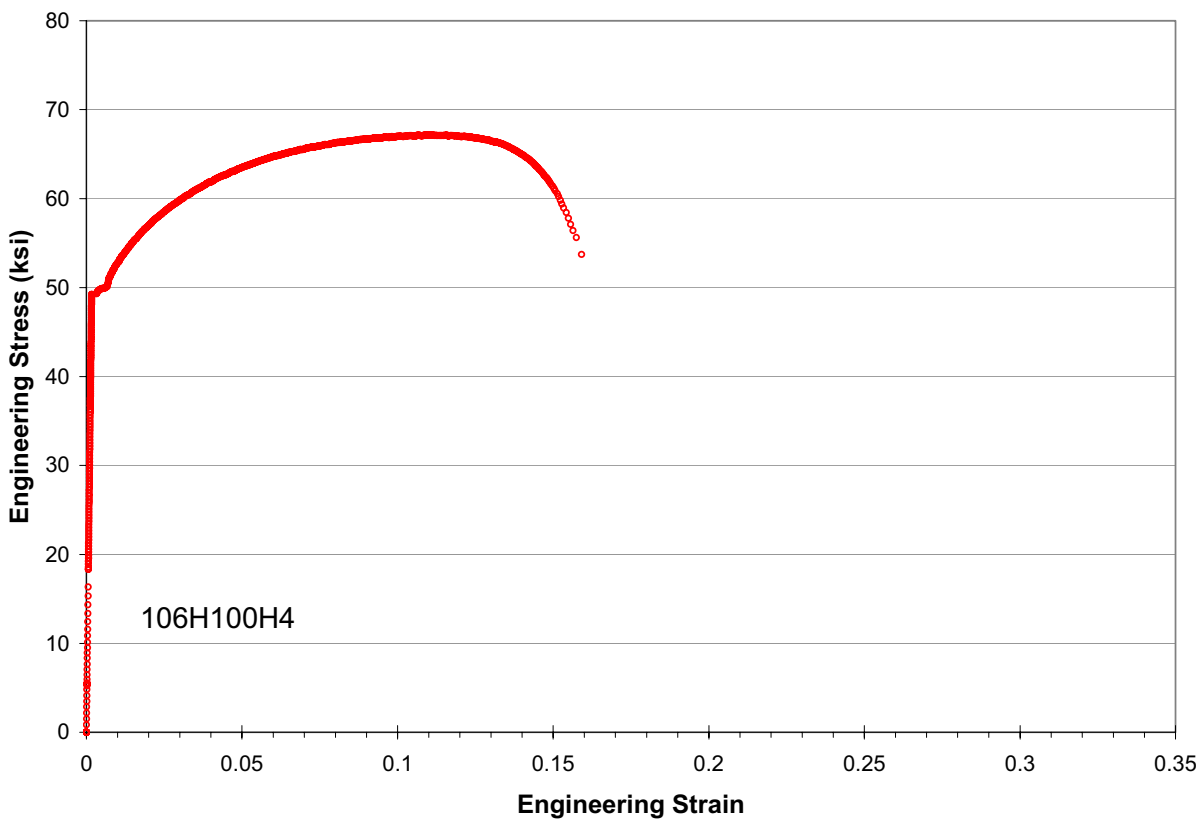


Figure 40: Engineering Stress vs. Strain for Sample # 106H100H1 (HAZ in 1500 psig H₂)

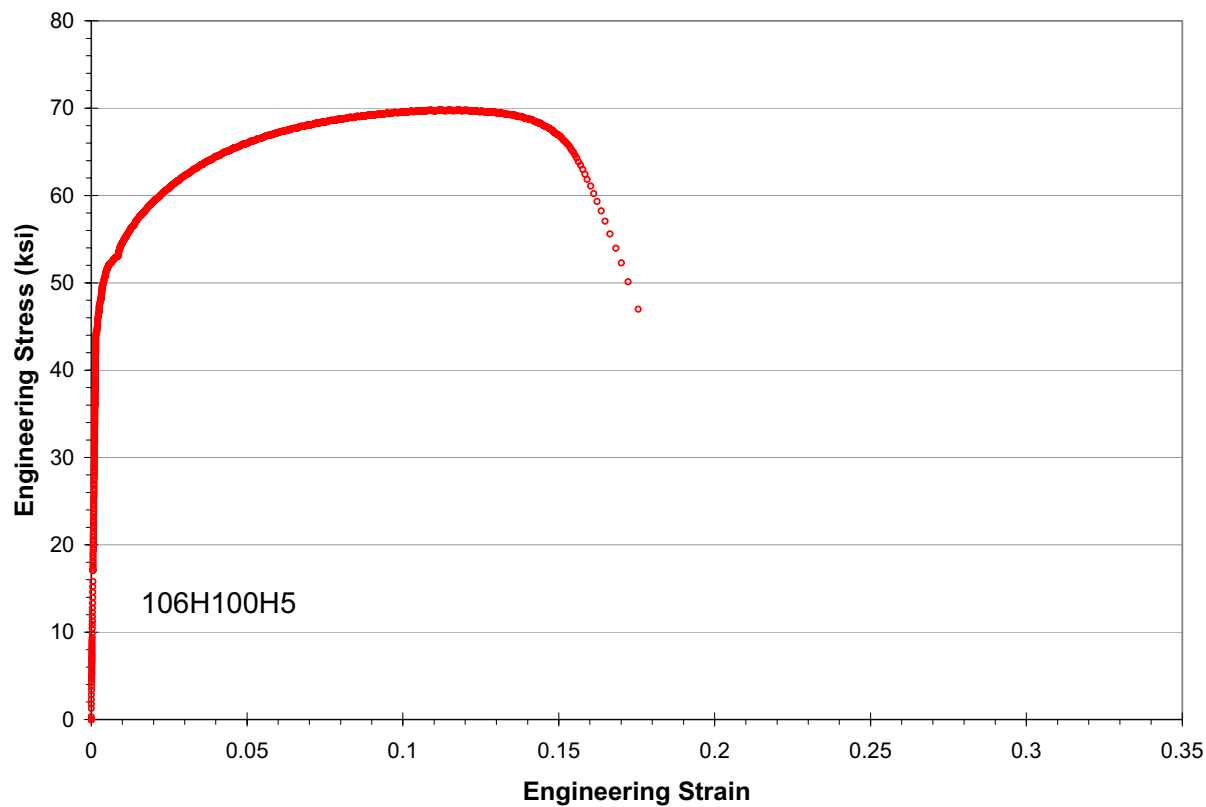


Figure 41: Engineering Stress vs. Strain for Sample # 106H100H1 (HAZ in 1500 psig H₂)

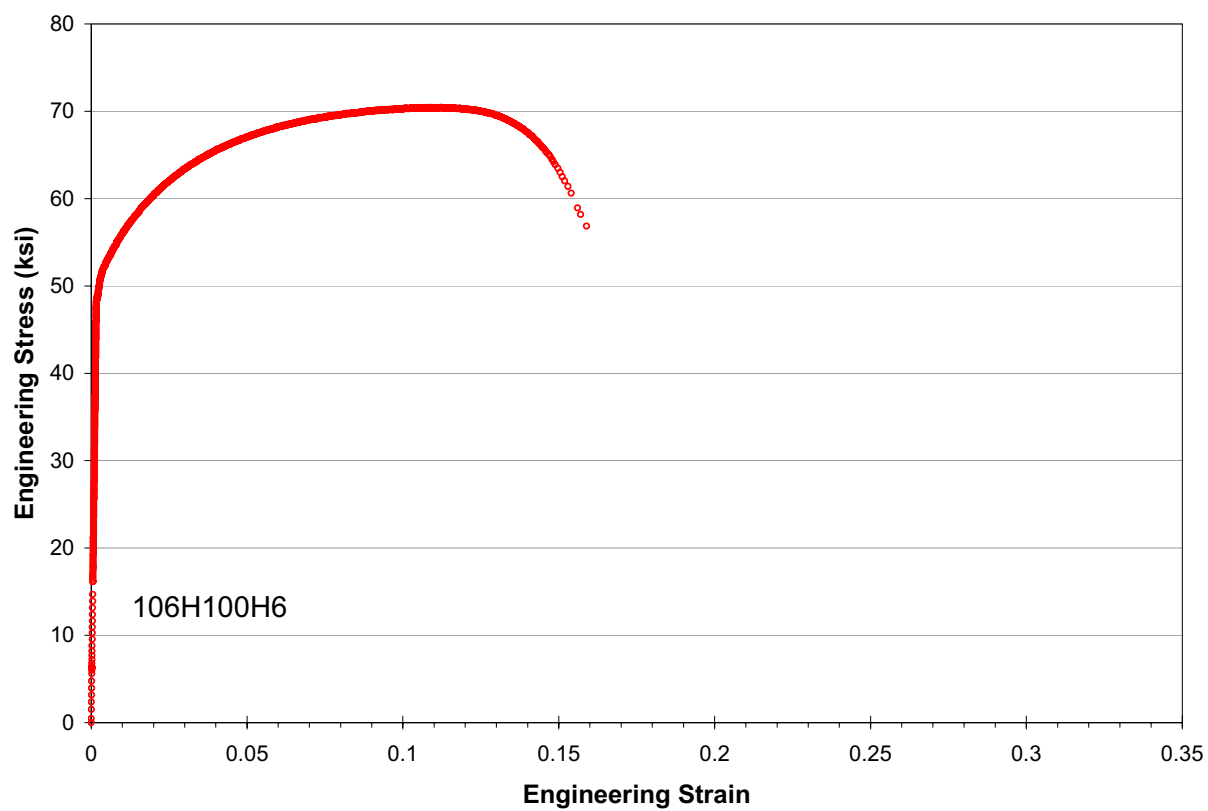


Figure 42: Engineering Stress vs. Strain for Sample # 106H100H1 (HAZ in 1500 psig H₂)

Table III: Tensile Property Results

	Sample	1 ATM (Air)			Sample	100 ATM (H2)		
		Yield (ksi)	UTS (ksi)	Elong. at failure		Yield (ksi)	UTS (ksi)	Elong. at failure
Base Metal	106B001A1	52.2	69.7	0.343	106B100H1	58.1	72.2	0.159
	106B001A3	53.2	69.5	0.248	106B100H2	48.1	69.6	0.157
	106B001A5	47.9	68.8	0.343	106B100H3	47.5	68.3	0.249
	106B001A7	51.8	71.7	0.238	106B100H4	56.6	71.1	0.170
	106B001A8	54.2	71.9	0.296	106B100H5	52.9	74.5	0.174
	106B001A9	49.9	70.1	0.300	106B100H6	47.6	67.6	0.225
	AVERAGE	51.5	70.3	0.29		51.8	70.6	0.19
	STDEV	2.3	1.3	0.04		4.8	2.6	0.04
Weld Metal	106W001A1	50.7	70.5	0.288	106W100H1	47.4	72.3	0.245
	106W001A2	46.5	70.1	0.261	106W100H2	52.1	69.3	0.231
	106W001A4	50.8	71.3	0.283	106W100H3	53.2	71.2	0.220
	106W001A6	49.9	71.4	0.292	106W100H4	49.1	69.1	0.191
	106W001A7	46.5	70	0.297	106W100H5	49.9	69.2	0.207
	106W001A8	54.1	73.5	0.281	106W100H6	52.9	67.4	0.182
	AVERAGE	49.8	71.1	0.28		50.8	69.8	0.21
	STDEV	2.9	1.3	0.01		2.3	1.7	0.02
HAZ	106H001A1	51.2	68.2	0.25	106H100H1	45.9	68.7	0.224
	106H001A2	56.1	71.1	0.213	106H100H2	51.1	70.4	0.156
	106H001A3	51.4	69.6	0.279	106H100H3	45.6	67.6	0.261
	106H001A4	48.2	69.4	0.315	106H100H4	49.7	67.1	0.159
	106H001A5	48.5	71.0	0.297	106H100H5	50.0	69.7	0.176
	106H001A6	48.6	70.4	0.236	106H100H6	52.0	70.3	0.159
	AVERAGE	50.7	70.0	0.27		49.1	69.0	0.19
	STDEV	3.0	1.1	0.04		2.7	1.4	0.04

5.0 SUMMARY AND CONCLUSIONS

This study reports the interim results from the testing program to support the evaluation of the use of new and existing pipelines and systems to carry and to deliver hydrogen as an alternative energy source under the hydrogen economy. The carbon steels alloy A106 Grade B was selected because of its common application in pipelines. The results of a matrix of tensile tests performed at room temperature in air and in high pressure hydrogen were reported.

A general reduction of the ductility or deformation capacity was noted in this material. The elongation to failure and reduction in area was reduced when specimens were tested in 1500 psig hydrogen. Furthermore, the primary mode of fracture changed from ductile rupture to cleavage.

Tests to quantify the fracture behavior in terms of J-R curves for these materials at air and hydrogen pressure conditions are being planned.

6.0 ACKNOWLEDGEMENTS

The technical input of Thad Adams, Glenn Chapman, Mike Morgan and Joel Jones toward the development of the experimental approach is gratefully acknowledged. Technical review of this manuscript was performed by Thad Adams.

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