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THE EFFECT OF HYDROGEN AND TEMPERATURE ON MECHANICAL PROPERTIES OF THE Ti-5Al-2.5Sn ELI ALLOY

UNCLASSIFIED NERVA RESEARCH
AND DEVELOPMENT REPORT.

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I. SUMMARY

The effects of hydrogen content on the mechanical properties of the Ti-5Al-2.5Sn ELI alloy were investigated in the temperature range -320 to +200°F. In addition, the absorption of hydrogen by the alloy was studied to determine safe operating limits for the 0.020-inch thick core band.

The data show that Ti-5Al-2.5Sn ELI can tolerate 300 ppm hydrogen at all temperatures investigated. This is twice the hydrogen content permitted by WANL specifications.

It was established that the core band can safely be heated in hydrogen to 750°F for 3 hours and to 650°F for at least 5 hours during decay heating in NERVA reactors. The effects of pressure and radiation on the absorption of hydrogen during reactor operation were judged to be negligible.

II. INTRODUCTION

Titanium and its alloys are subject to embrittlement when hydrogen contents are sufficiently high to precipitate titanium hydride which acts as an internal stress raiser. The rate of hydrogen absorption and the minimum hydrogen content for embrittlement vary with composition. The rates of absorption and diffusion are also dependent on temperature and surface condition. For example, the thin oxide film formed on titanium surfaces upon exposure to the earth's atmosphere is an effective barrier to the diffusion of gaseous hydrogen.[3]

On the basis of data then available[2] for unalloyed titanium, the following conclusions were reached for the Ti-5Al-2.5Sn ELI (extra-low interstitial content) alloy:

1. The specification limit of 150 ppm hydrogen would not seriously affect mechanical properties.
2. Appreciable absorption of hydrogen would not occur below 250°C (942°R), which is well above the maximum temperature seen by the dome end support ring.
3. High pressures of hydrogen would not enhance absorption and diffusion of the gas because these processes are inoperative at low temperatures.
4. The amount of hydrogen absorbed through radiation effects would be negligible.

Since the addition of aluminum and tin to titanium increases the solubility of hydrogen in alpha titanium[4-7] (thus decreasing the possibility of hydride formation), these conclusions[2] are conservative.

Alpha titanium is the hexagonal, close-packed allotrope of unalloyed titanium which exists below 1620°F. The combination of 5 per cent aluminum and 2.5 per cent tin is soluble in alpha titanium and, for the ELI grade, increases to 1675-1725°F the range in which alpha is stable. Above 1620°F, unalloyed titanium transforms to body-centered-cubic beta, which is stable to the melting point. Ti-5Al-2.5Sn ELI exists as alpha plus beta from about 1675 to 1875°F, above which it is all beta to the melting point. Hydrogen, however, stabilizes the beta phase and lowers the allotropic transformation temperatures.

Since the publication of the previous study,[2] data from three investigations on the effect of hydrogen on Ti-5Al-2.5Sn (but not the ELI grade) have been found in the literature. Day[7] showed that contents of up to 400 ppm slightly increased the notched (K_t = 3) and unnotched tensile strengths of 0.040-inch thick sheet, but did not affect elongation and the notched-to-unnotched tensile strength ratio at room temperature and -65°F. Riesen and Kah[8] found that concentrations of up to 312 ppm hydrogen slightly increased room-temperature tensile strengths, but did not appreciably affect notched strengths (K_t = 4.2) or the stress to rupture notched specimens in 100 hours, also at room temperature.

Haynes[1] conducted an extensive evaluation of room-temperature mechanical properties at concentrations up to 900 ppm hydrogen. Embrittlement was found to occur above about 360 ppm in Izod impact, tensile, notched tensile (K_t = 3.9), and notched stress-rupture tests. Since the present investigation paralleled Haynes' work, much of his data are included this report.

This investigation was conducted for several purposes. First, all previous work was conducted on non-ELI material. Since the ELI grade has less iron and oxygen than the normal commercial grade of Ti-5Al-2.5Sn, the effects of these compositional differences required evaluation. Secondly, there were no data on the effects of hydrogen

at temperatures other than at room temperature. The possibility that hydrogen could have more severe effects on cryogenic or elevated temperature properties meant that hydrogen limits might have to be reduced for NERVA parts. Finally, the 0.020-inch-thick core band can be heated to 1000°R during reactor operation; this is close to the temperature at which hydrogen absorption and diffusion rates become appreciable, and time and temperature limits for the core band had to be established in the absence of these data from the literature.

III. EXPERIMENTAL PROCEDURES

A. Materials

Three forms of commercially-produced material were procured to WANL specifications for Ti-5Al-2.5Sn ELI. One-half-inch diameter bar was used for notched and unnotched tensile tests, and for notched stress-rupture tests; 0.5-inch-square bar was used for Charpy impact tests; and 0.020-inch-thick sheet was used to determine the limiting times and temperatures for core band operation.

Compositions of these materials are given in Table I together with the composition of Haynes' 0.625-inch-diamter bar,[1] and, for comparison purposes, specification limits for the normal and ELI grades of Ti-5Al-2.5Sn.

The materials used for this study were quite uniform in composition, and none of the small differences noted would be expected to affect test results. Haynes' material, however, had a significantly lower iron content and higher oxygen and carbon contents, but his oxygen is typical of non-ELI Ti-5Al-2.5Sn.

B. Hydrogenation

The hydrogenation of bar specimens was done prior to machining to test configuration. Test coupons in batches of six were degassed and then ingassed at 1350°F to the desired hydrogen content using a Sievert's apparatus modified to perform both operations. After hydrogenation, each sample was homogenized at 1350°F for 16 hours under a continuous flow of purified argon. Control specimens were degassed in the Sievert's apparatus and subjected to the same thermal treatment, after which all specimens were machined to their final configurations. After testing, samples from representative specimens were analyzed and the reported data are based on actual, rather than nominal, hydrogen contents. Haynes[1] degassed his specimens for 16 hours and then hydrogenated at 1475°F for 4 hours.

To determine the limiting time and temperatures for operation of the titanium alloy core band in hydrogen, machined tensile specimens of 0.020-inch-thick sheet, were racked in an Inconel fixture which was constructed to allow the free circulation of hydrogen. About eight specimens, with 0.25-inch wide, one-inch-long gage sections were hydrogenated at one time. Notched sheet tensile specimens (1-inch major width, 0.5-inch minor width, 0.005-inch root radius, and 6.1 K_t) were similarly treated. Hydrogenation was performed in an electrically-heated furnace under a flow of dried and deoxidized hydrogen gas.

C. Mechanical Testing

Unnotched tensile tests of bar material were conducted on standard subsize specimens with 0.250-inch-diameter gage sections. Elongation measurements were made over a length equal to four diameters (1 inch). Haynes[1] measured elongation over a length equal to four times the square root of the cross-sectional area, which is equivalent to measuring over a length of 3.54 diameters; since most of the elongation is confined to the area adjacent to the fracture, Haynes' elongation values would tend to be somewhat larger than those obtained in this work.

The strain rate used in unnotched tensile tests of sheet and bar was 0.005 in/in/min to fracture. This is compared in Table II with the strain rates used by Haynes.

Bar specimens for notched tensile and notched stress-rupture tests had a major diameter of 0.250 inch and contained a central circumferential notch which reduced the cross-sectional area by one-half. The root radius was 0.005 inch and the theoretical notch concentration factor, K_t, was 3.9. Notched tensile tests were conducted at a constant cross-head speed of 0.005 in/min. Haynes' notched specimens were identical,[1] but he did not report a rate of straining or loading.

All tensile tests were conducted on a screw-driven testing machine using a deflectometer on the moving cross-head to measure strain. Experience has demonstrated

that, on this machine, deflectometer measurements are essentially equivalent to extensometer measurements on the specimen size used in this study.

Notched-stress rupture tests were performed on lever-type creep machines of standard construction. Loads were based on a percentage of the unnotched tensile strength and were imposed for 100 hours, unless the specimen fractured within this period. Specimens surviving the 100-hour test were retested at increased stresses until failure occurred within 100 hours at the final stress.

Impact testing was performed on V-notch Charpy specimens of standard design on a recently calibrated machine.

D. Metallography

Surface preparation of the samples was done using grinding and polishing techniques which are conventional for the Ti-5Al-2.5Sn alloy. Etching was performed in two steps. A dull etch (consisting of 35 per cent by volume of concentrated nitric acid, 35 per cent hydrogen peroxide, 5 per cent concentrated hydrofluoric acid, balance distilled water) was used to remove worked metal and scratches. A final, bright etch was then used to delineate microstructural details. This final etch consisted of 3 per cent by volume concentrated nitric acid, 2 per cent concentrated hydrofluoric acid, balance distilled water.

IV. EXPERIMENTAL RESULTS AND DISCUSSION

A. Effect of Time and Temperature on Hydrogen Absorption

These effects are reported in Table III. No absorption was observed after exposure to hydrogen for 5 hours at 575 and 650°F (1035 and 1110°R) and after exposure at 750°F (1210°R) for 3 hours. Hydrogen absorption occurred during exposure for 5 hours at 750°F and during briefer exposures at 850 and 925°F. The relatively low hydrogen contents shown for 650°F (5 hours) and 750°F (3 hours) can be attributed to slight variations in hydrogen in the starting material and, possibly, analytical techniques.

The fact that an incubation period of between 3 and 5 hours at 750°F was required to initiate hydrogen absorption can be explained on the basis of the existence, on the sheet surfaces, of a coherent oxide film which dissolves slowly in the metal at and below this temperature. The film is formed naturally by the reaction of the metal with the earth's atmosphere, and is present on all titanium components of NERVA reactors. Film formation occurs in minutes or less upon exposure of clean (e.g., chemically etched) surfaces to the air. Katz and Gulbransen[3] have shown for the zirconium-hydrogen system, which is exactly comparable with the titanium-hydrogen system, that oxides formed at room temperature have the greatest effect on the rate of hydriding. Films formed at higher temperatures are less resistant to hydriding because film thickness is actually less due to the increased solubility of oxygen at elevated temperatures, and because these films are more porous than room-temperature films. Complete removal of the film (by solution of the oxide during vacuum and annealing) was shown to result in instantaneous absorption at an extremely high rate.[3]

Specimens exposed to hydrogen at 750°F (5 hours), 850°F, and 925°F were thoroughly hydrided, and disintegrated in the furnace from stresses arising from expansion of the atomic lattice by absorbed hydrogen atoms. All other specimens were

tensile tested, but the data are not reported since, as could be predicted, properties were identical to those of the as-received material.

No attempt was made to determine the effect of pressure on the rate of hydriding. However, the hydrogen pressures seen by the core band during start-up and operation should not affect the integrity of the part because absorption rates are negligible at the temperatures involved (about 140°F and below). During decay heating, pressures are negligible, so that the data in Table III accurately indicate safe operating limits. Although microstructures and mechanical properties have not yet been evaluated for tested NERVA reactors, the behavior, during disassembly, of the NRX-A2 and A3 core bands indicates that significant hydrogen embrittlement did not occur.

B. Effect of Radiation on Hydrogen Absorption

In a previous study by WANL,[2] it was conservatively calculated that the radiation-induced absorption of hydrogen in the thinnest (0.120 inch) section of the dome end support ring would be two orders of magnitude below the maximum concentration permitted by WANL specifications. Using a somewhat different approach, Dixon[11] of Aerojet-General Corporation arrived at a similar estimate of the effect of radiation. Furthermore, he determined that the small amount of absorbed hydrogen would not penetrate deeper than 0.00003 inch into the titanium alloy. Although the core band operates at a higher temperature than the support ring, the rate of radiation-induced absorption would not increase significantly, because the mechanism is temperature-insenstitive. Therefore, the effects of radiation on hydrogen pickup and on the subsequent properties of the material can be disregarded.

C. Effect of Hydrogen on Tensile Properties

These data are presented and compared with Haynes' room temperature data[1] in Figures 1-6. Comparison of Haynes' data with the room-temperature data

generated in this investigation is complicated by differences in strain rates and composition, as shown in Tables II and I, respectively.

Furthermore, Haynes heated his specimens at 1475°F, 125°F higher than the temperature used during the present work. Finally, significant differences in fabrication and test techniques may exist between the two investigations.

The higher carbon content of Haynes' material, shown in Table I, has been shown to increase, by about 10,000 psi, the 0.2 per cent yield and ultimate tensile strengths of high purity titanium, and to reduce elongation by about 3 per cent (absolute).[9, 10] The higher oxygen content of Haynes' alloy over that of the ELI grade has also been shown to increase the strength of pure titanium by 10,000 psi and to reduce elongation by 6%.[9, 10] The net effect of increased oxygen and carbon would be roughly additive. The lower iron content of Haynes' material could be expected to reduce strengths slightly (3-4000 psi) and to increase elongation by about 4 per cent.[9]

Because of the strain-rate sensitivity of titanium, the yield strengths obtained in this investigation could be predicted to lie between Haynes' low and high strain rate data, and ultimate tensile strengths and ductility values could be predicted to approximate those obtained by Haynes at the lowest strain rate (assuming no differences in composition, heat treatment, and fabrication and test techniques).

Consideration of all of the processing, testing, and compositional differences between the two investigations would lead one to predict the following differences in the room-temperature tensile test data for given hydrogen contents.

1. Ultimate Tensile Strength: Haynes' data would be about 15,000 psi higher at the low strain rate and about 25,000 psi higher at the high strain rate.

2. 0.2% Yield Strength: Haynes' data would be about 12,000 psi higher at both strain rates.
3. Elongation: Haynes' data would be lower by about 6 per cent (absolute) at the low strain rate and about 10% lower at the high strain rate. However, since his measurements were taken over a shorter length, elongations could approximate those obtained in this work.
4. Reduction in Area: Absolute effects could not be estimated, but values would be estimated to be lower than those of the present investigation.
5. Notched Tensile Strength: Haynes' data would be comparable to data obtained in this study.

As shown in Figure 1, the ultimate tensile strengths obtained at room temperature paralleled those obtained by Haynes, but were slightly higher. The reason for the observed deviation from predicted values is not known, but could possibly be attributed to unknown differences in fabrication and/or testing. However, it is more likely that microstructural differences are responsible for the phenomenon.

Haynes reported that hydrogen in excess of 350 ppm depressed the alpha transus (the temperature above which alpha begins to transform to, and coexists with beta) below the annealing temperature of 1475°F, thus enabling beta to form at alpha grain boundaries.[1] In this investigation, beta was first observed at about 150 ppm hydrogen and it was found at the grain boundaries, but mostly within the grains, Figure 9(b). This discrepancy is discussed in detail in other sections of this report, but it can be explained on the basis of the ten-fold difference in iron content, noted in Table I, which depressed the alpha transus to a greater extent in the ELI material, and which preferentially dissolved hydrogen to produce larger quantities of beta phase in the microstructure of the ELI alloy for given hydrogen contents. Since iron strengthens titanium,

the higher strength of the ELI bar can be explained on this basis. At elevated temperatures, beta is well known to be detrimental to strength, as demonstrated by the decrease in strength which occurred at 200°F at hydrogen contents in excess of about 200 ppm.

Below room temperature, hydrogen is shown to be an effective strengthener. Strength increases of up to 20,000 psi were observed at the 300-400 ppm level, but strengthening effects appear to be peaking out at this level.

The yield strengths obtained in this investigation (Figure 2) paralleled the trends noted for ultimate tensile strengths and are explicable on the same bases. Haynes reported data only for the 0.1% offset yield strength, but the data can be compared directly with this investigator's, since the 0.1% and 0.2% yields were observed to be almost identical. The decrease in Haynes' yield strengths above about 360 ppm hydrogen coincided with the presence of substantial quantities of hydride in the microstructure. Because of compositional differences, hydride was not observed in the ELI alloy below the 475 ppm level, and the quantity of hydride present evidently was insufficient to adversely affect the yield strength. Haynes attributes the drop in yield strength (and proportional limit) to non-uniform deformation of alpha, induced by hydride stress-risers, at mean stresses below the elastic limit of hydride-free material.[1]

The room-temperature ductility values determined in the two investigations showed similar trends (Figures 3 and 4). The increase in ductility which occurred with increasing hydrogen content can be attributed, at least in part, to increased proportions of hydrogen-stabilized, body-centered-cubic beta, which is more ductile than hexagonal alpha titanium. When hydride precipitated, ductility values decreased. At cryogenic temperatures, ductility was unaffected by hydrogen contents of up to 400 ppm.

The room-temperature notched tensile strengths from both investigations (Figure 5) were comparable. Notched strengths fell off as hydride became visible in the microstructure. At -100°F, hydrogen had a slight strengthening effect, but the notched

strength at -320°F remained constant up to hydrogen contents of between 300 and 450 ppm where a drastic reduction in strength was observed. Based on metallographic observations, hydride probably did not appear until the 400 ppm level was reached, so that the curve for the -320°F data is probably conservatively drawn.

The notched:unnotched tensile strength ratios are plotted in Figure 6. Although the ratios for Ti-5Al-2.5Sn ELI decreased continually with hydrogen content, values were still respectably high (1.55) at room temperature for 450 ppm hydrogen. The ratios at 300 ppm hydrogen were 1.45 and 1.40 for -100 and -320°F, respectively.

D. Effect of Hydrogen on the Notched Stress-Rupture Limit

Data of the present investigation for the 100-hour stress-rupture limit are compared with Haynes' data for a 24-hour limit in Figure 7. Since the limit is time-dependent, the 100-hour data were expected to be below the 24-hour data; however, the actual values obtained are quite comparable. The data obtained for the ELI grade confirm Haynes' conclusion[1] that 300 ppm hydrogen is a reasonable limit for the Ti-5Al-2.5Sn alloy.

E. Effect of Hydrogen on Impact Properties

Despite the noted differences in composition, heat treatment, and test method, this author's data compare favorably with those of Haynes (Figure 8). Furthermore, sharp reductions in impact values occurred at about the same hydrogen level, 350 ppm, at room and cryogenic temperatures. Impact values for 200°F tests remained at relatively high levels up to 550 ppm because of the increased solubility of hydrogen at this temperature.

F. Effect of Hydrogen on Microstructure

Microstructures of the fractures of the notched stress-rupture specimens are presented in Figure 9. As indicated previously, Haynes observed beta phase to be present at grain boundaries at and above 350 ppm hydrogen, and beta phase became evident in the ELI grade (principally within the grains) at 150 ppm hydrogen, Figure 9(b). This was attributed to a ten-fold difference in iron content, as shown in Table I, which had a larger depressive effect on the alpha transus of the ELI material. Iron, in concentrations shown for the ELI grade, appears as discrete particles within the alpha grains (the dark dots in Figure 9(a) are iron-stabilized beta), but at extremely low concentrations, all of the iron may be concentrated in the grain boundaries. When hydrogen is absorbed by the beta phase, the proportion of beta-to-alpha increases, and the increases are manifested as larger particles at the sites of the beta which existed prior to hydrogenation. This explains why Haynes' beta phase was concentrated at the grain boundaries, while beta in the ELI grade was principally intragranular (a thin envelope of beta could be observed under polarized light in ELI specimens containing more than about 150 ppm hydrogen.)

Haynes[1] also reported the presence of hydride at hydrogen levels of 300 ppm and greater. He also found hydride to occur exclusively at grain boundaries. In this study, evidences of hydride were not detected until the 475 ppm level was reached, and the hydride was generally found within the grains and only occasionally at grain boundaries. Since hydride precipitates from beta upon cooling from the hydrogenation temperature, because of a decrease in solubility at low temperatures, the location of the hydride plates is understandable. The hydrides observed in this study are shown in Figure 9(f), where they appear as white plates under polarized light.

The fact that Haynes observed hydride at lower hydrogen contents than did this investigator is attributable to the increased solubility of hydrogen in the ELI material -- a result of the higher iron content. Since hydrides can be precipitated from beta titanium and result in embrittlement during slow-strain-rate tensile and stress-rupture

tests, one might have predicted that the ELI material would be embrittled at lower hydrogen levels than Haynes' material. However, the ELI material contained more beta phase for given hydrogen contents, thus reducing the concentration of hydrogen in beta and decreasing the possibility of embrittlement. Since hydrogen preferentially partitions to beta, the alpha phase of Haynes' low-iron material had a larger concentration of hydrogen for a given hydrogen content; consequently, the solubility limit for hydrogen in alpha was achieved sooner in Haynes' Ti-5Al-2.5Sn, and hydride plates became evident at hydrogen levels which were relatively low compared to those for the Ti-5Al-2.5Sn ELI. Any analysis of the effects of the discrepancy in iron content is necessarily complicated by other compositional discrepancies, differences in hydrogenation temperatures, and, possibly, differences in raw material fabrication and test techniques. However, the net result was that embrittlement of the two materials occurred at comparable hydrogen contents.

Microstructures of the fractures of the notched stress-rupture specimens are shown in Figure 9. The increase in beta (dark etching constituent) with hydrogen content is shown in (a) through (d). The beta is oriented parallel to the direction of rolling, which is not unusual for this alloy.

One would not expect beta phase to be present in commercially-annealed Ti-5Al-2.5Sn ELI to the extent shown in Figure 9. This is so because the cooling rate from the mill annealing temperature is much slower than the rate used in this investigation. The cooling rate obtained in commercial practice allows beta to transform to alpha as the temperature is reduced, thereby resulting in essentially equilibrium structures. In this investigation, cooling rates were relatively rapid, and some portion of the beta shown in Figure 9 is undoubtedly metastable. In commercial Ti-5Al-2.5Sn ELI, therefore, more alpha phase will be present for a given hydrogen content than was in this investigator's material. Although the solubility of hydrogen in alpha is less than it is for beta, the increased proportion of alpha in commercial material over that of the experimental material

of this study would have increased capabilities for retaining a larger total number of hydrogen atoms in solid solution. On this basis, it is estimated that the tolerance of the Ti-5Al-2.5Sn ELI alloy for hydrogen will not be appreciably altered by the described variations in microstructure.

Fractures were transgranular up to 288 ppm hydrogen, but became -- at least in part -- intergranular at 473 ppm. The mode of fracture at the 473 ppm level is more evident in Figure 9(e). The hydride plates, which are shown as they appear under polarized light in Figure 9 (f), are seen to occur mainly within the grains; however, the plates oriented 90° to each other in the upper right-hand corner are in grain boundaries.

V. CONCLUSIONS AND RECOMMENDATIONS

A. The core band will not be embrittled by hydrogen during decay heating for up to 3 hours at 750°F (1200°F) and for at least 5 hours at 650°F (1100°R).

B. High hydrogen pressures during reactor operation should not embrittle the core band since temperatures are too low for the initiation of absorption and diffusion of hydrogen.

C. The effect of radiation on hydrogen absorption is negligible.

D. Unnotched tensile properties of the Ti-5Al-2.5Sn ELI alloy in the range -320°F to +200°F are not adversely affected by hydrogen contents of up to 400 ppm.

E. Notched tensile properties from -100°F to +200°F are unaffected by hydrogen contents of up to 400 ppm. However, embrittlement during tests at -320°F occurs at the 350-400 ppm level.

F. Notched stress-rupture and tests indicate that about 300 ppm hydrogen can safely be tolerated by the alloy.

G. Impact embrittlement of Ti-5Al-2.5Sn ELI occurs at the 350-400 ppm level from room temperature to -320°F.

H. The present WANL specification limit of 150 ppm for the Ti-5Al-2.5Sn ELI alloy is conservative. However, no relaxation of this requirement is suggested since this will not result in a cost reduction, and it will reduce the margin of safety if core band temperatures increase above their present level.

VI. REFERENCES

1. R. Haynes, "Effect of Hydrogen on Room-Temperature Mechanical Properties of Titanium-5% Aluminum-2.5% Tin Alloy," Journal of the Institute of Metals, v. 90, pp. 80-84, 1961-1962.

2. A. Filippi, "Selection of Titanium for NRX-A Reflector Dome End Support Ring," Addendum to WANL-TME-240, August 8, 1963.

3. O. M. Katz and E. A. Gulbransen, "Occluded Gases in Transition Metals," Westinghouse Research Laboratories Scientific Paper 63-139-120-P2, March 18, 1963.

4. G. A. Lenning, L. W. Berger, and R. I. Jaffee, "The Effect of Hydrogen on the Mechanical Properties of Titanium and Titanium Alloys," Fourth Summary Report under Contract DA1-33-019-505-ORD-(P)-1, 1955.

5. G. A. Lenning, J. W. Spretnak, and R. I. Jaffee, "The Effect of Hydrogen on Alpha Titanium Alloys," Trans. AIME, v. 206, pp. 1235-1240, 1956.

6. L. W. Berger, D. N. Williams, and R. I. Jaffee, "Hydrogen in Titanium-Aluminum Alloys," Trans. AIME, v. 212, pp. 509-513, 1958.

7. Dwayne L. Day, "Mechanical Metallurgy of Titanium," presented at the Titanium Metallurgy Course at New York University, September 9-13, 1957.

8. A. E. Riesen and D. H. Kah, "Hydrogen Embrittlement of Titanium Alloys," WADD Technical Report 60-275, October, 1960.

9. W. L. Finlay and J. A. Snyder, "Effects of Three Interstitial Solutes (Nitrogen, Oxygen, and Carbon) on the Mechanical Properties of High-Purity, Alpha Titanium," Trans. AIME, v. 188, pp. 277-286, 1950.

10. R. I. Jaffee, H. R. Ogden, and D. J. Maykuth, "Alloys of Titanium with Carbon, Oxygen, and Nitrogen," Trans. AIME, v. 188, pp. 1261-1266, 1950.

11. C. E. Dixon, Aerojet-General Corporation, private communication.

TABLE I - COMPOSITION OF EXPERIMENTAL MATERIALS

Form	Al, %	Sn, %	Fe, %	O_2, %	N_2, %	H_2, %	C, %	Mn, %
0.020" sheet	5.2	2.5	0.16	0.07	0.014	0.011	0.025	0.002
0.5" dia. bar	5.4	2.5	0.18	0.105	0.01	0.0087	0.02	0.01
0.5" square bar	5.5	2.5	0.10	0.095	0.01	0.0102	0.03	0.02
0.625" dia. bar[a]	5.0	2.51	0.017	0.152	0.015	--	0.093	--
WANL Spec. for ELI Grade	4.70-5.60	2.00-3.00	0.25 max.	0.12 max.	0.05 max.	0.015 max.	0.08 max.	0.10 max.
AMS Spec. for Normal Grade	4.0 -6.0	2.0 -3.0	0.50 max.	0.20 max.	0.07 max.	0.003-0.020	0.15 max.	0.30 max.

[a] Haynes' experimental material[1]

TABLE II - STRAIN RATES FOR UNNOTCHED TENSILE TESTS

Test	Strain Rate
Present Investigation[a]	0.005 in/in/min through fracture
Haynes - Low Strain Rate	0.0002 in/in/min to 0.5% offset Y. S., then 0.003 in/in/min through fracture
Haynes - Low Strain Rate	0.01 in/in/min to 0.5% offset Y. S., then 1.0 in/in/min through fracture

[a] Strain rate for sheet and bar specimens.

TABLE III - ABSORPTION OF HYDROGEN BY Ti-5Al-2.5Sn ELI DURING EXPOSURE TO A CONSTANT FLOW OF GAS AT 1 ATMOSPHERE PRESSURE

Temperature, °F	Time, hrs.	Hydrogen Content, ppm
As received 0.020" sheet		120
200	1	150
575	1	150
575	3	150
575	5	145
650	5	70
750	3	105
750	5	8350
850	1	7750
925	3	15700

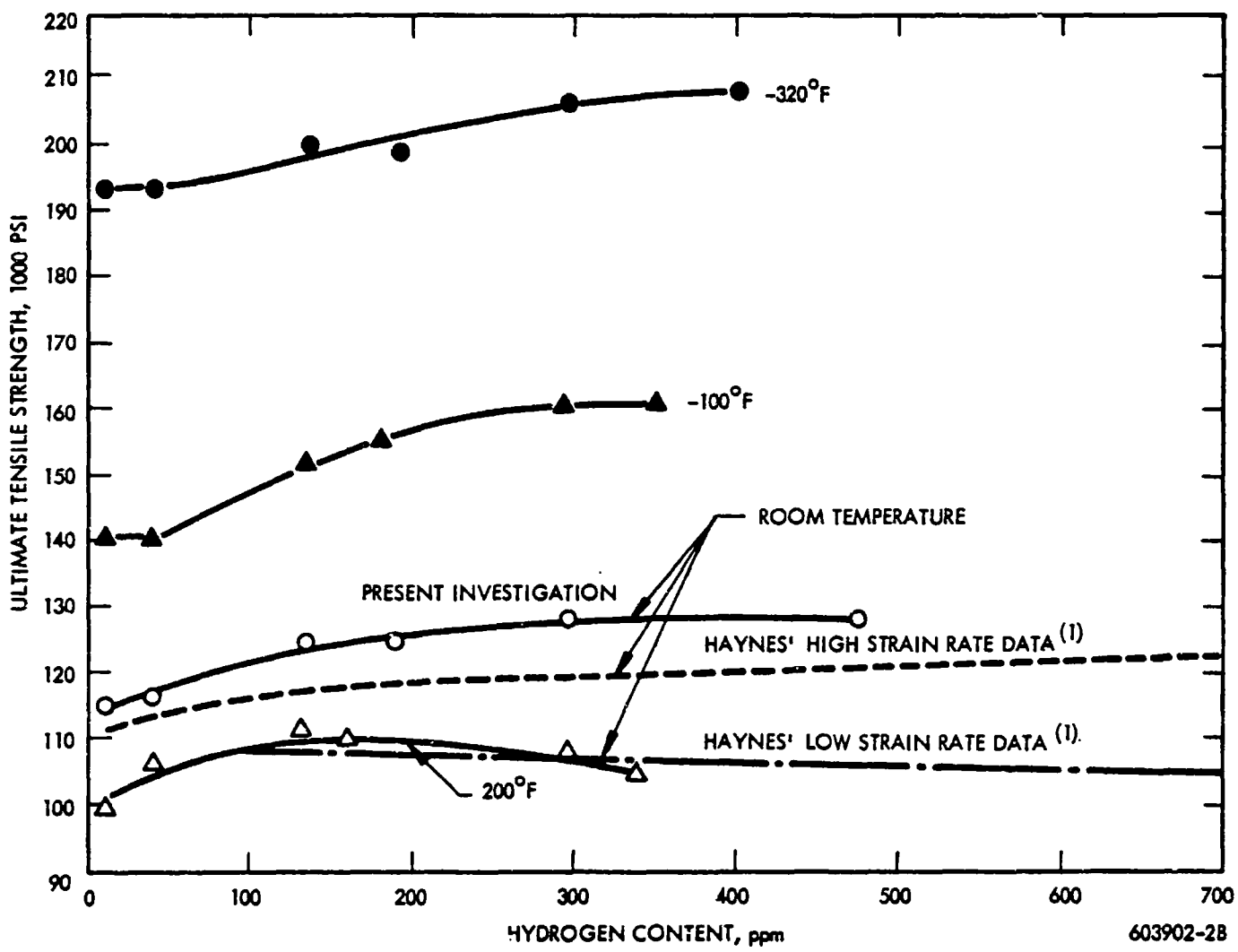


FIGURE 1 - The Effect of Hydrogen on the Ultimate Tensile Strength of Ti-5Al-2.5Sn

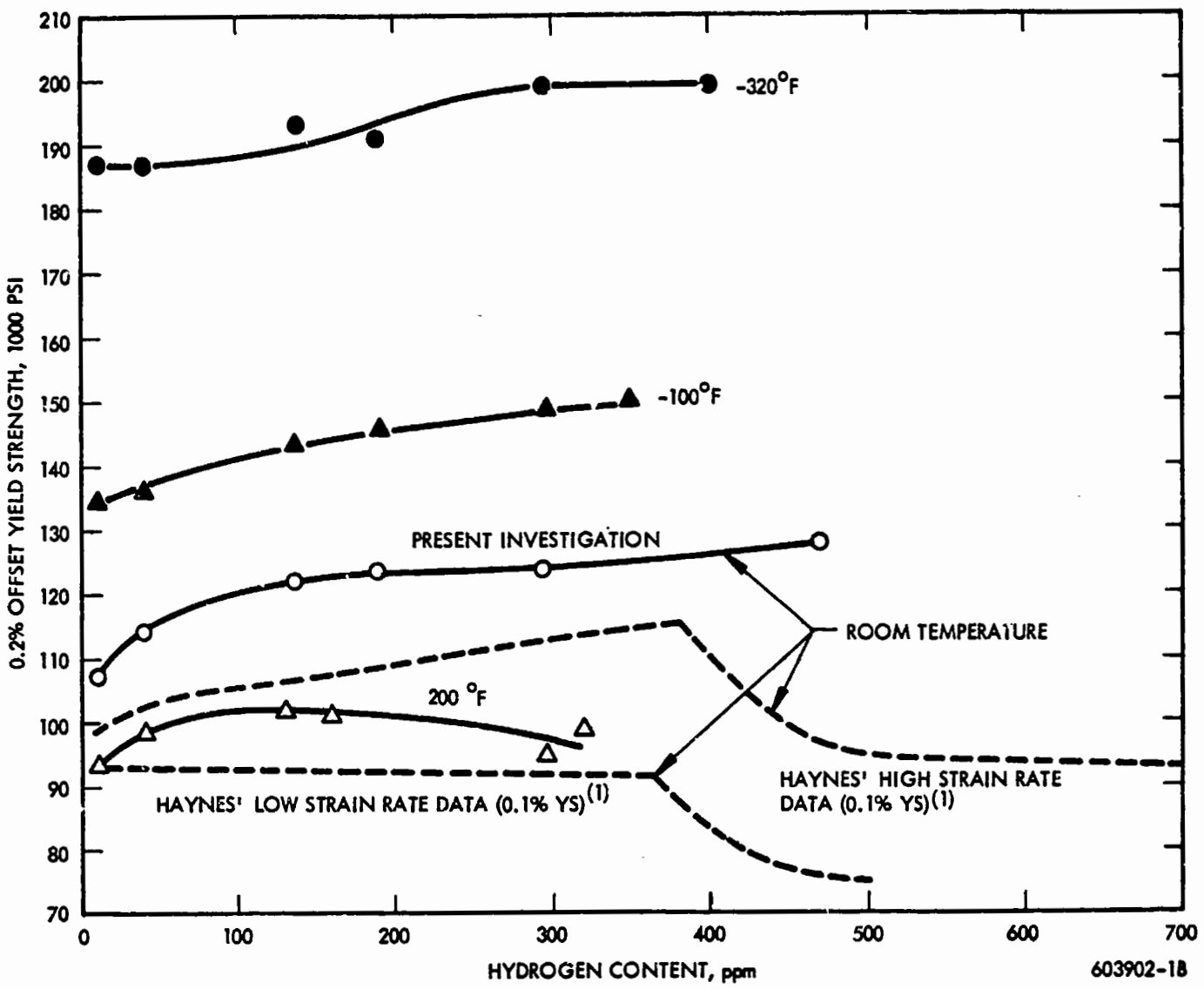


FIGURE 2 - The Effect of Hydrogen on the Yield Strength of Ti-5Al-2.5Sn

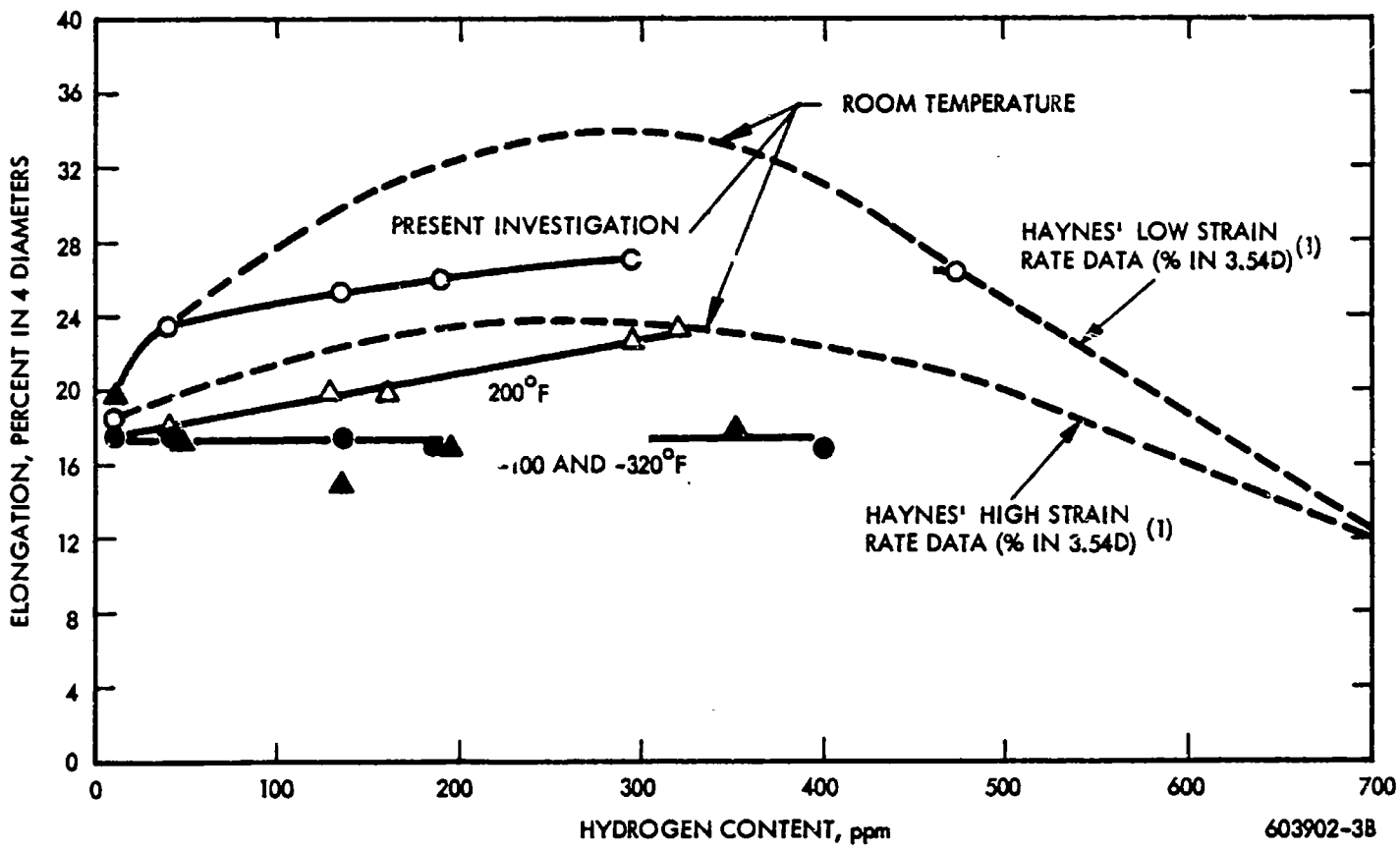


FIGURE 3 - The Effect of Hydrogen on the Elongation of Ti-5Al-2.5Sn

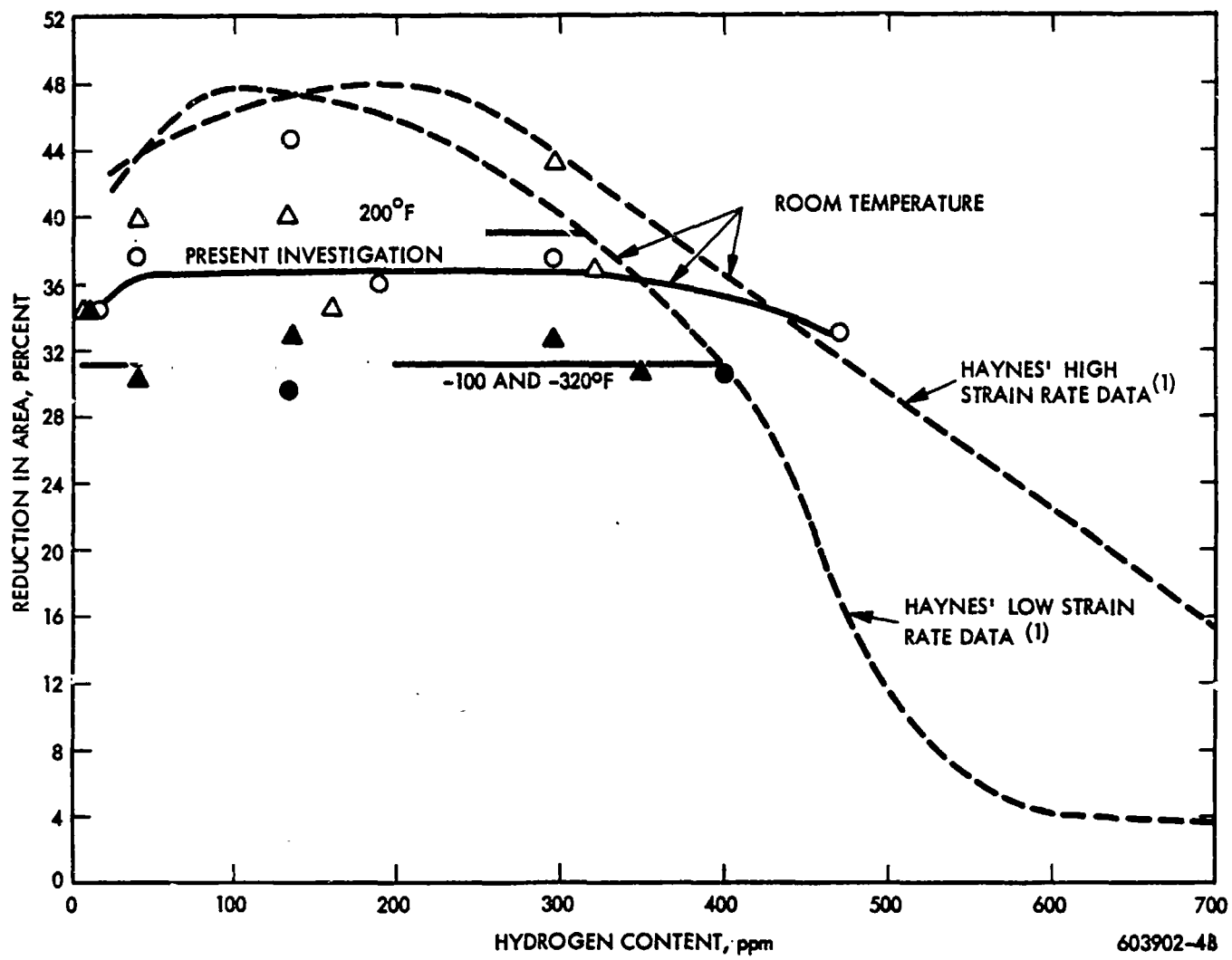


FIGURE 4 - The Effect of Hydrogen on the Reduction in Area of Ti-5Al-2.5Sn

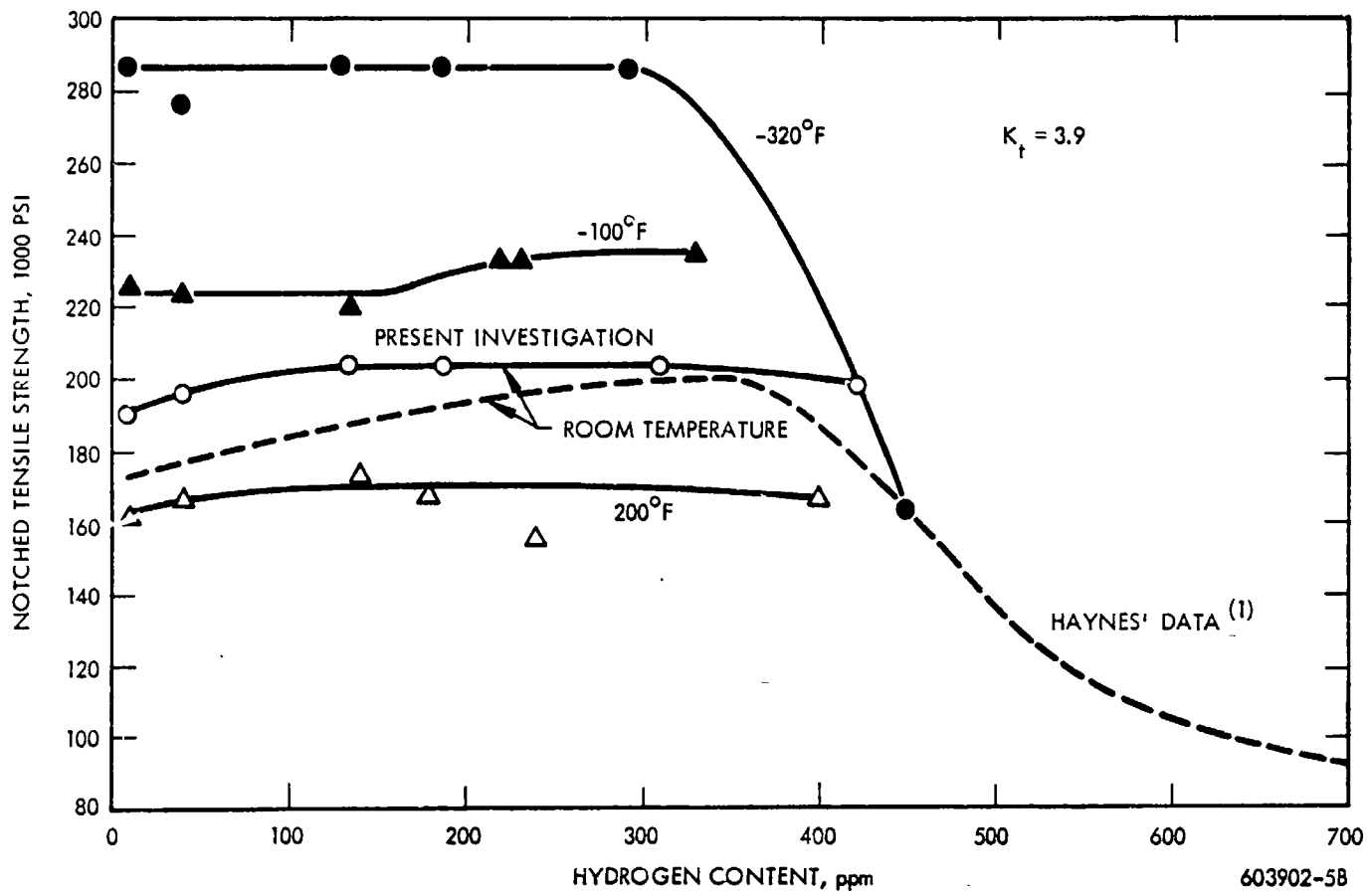


FIGURE 5 - The Effect of Hydrogen on the Notched Tensile Strength of Ti-5Al-2.5Sn

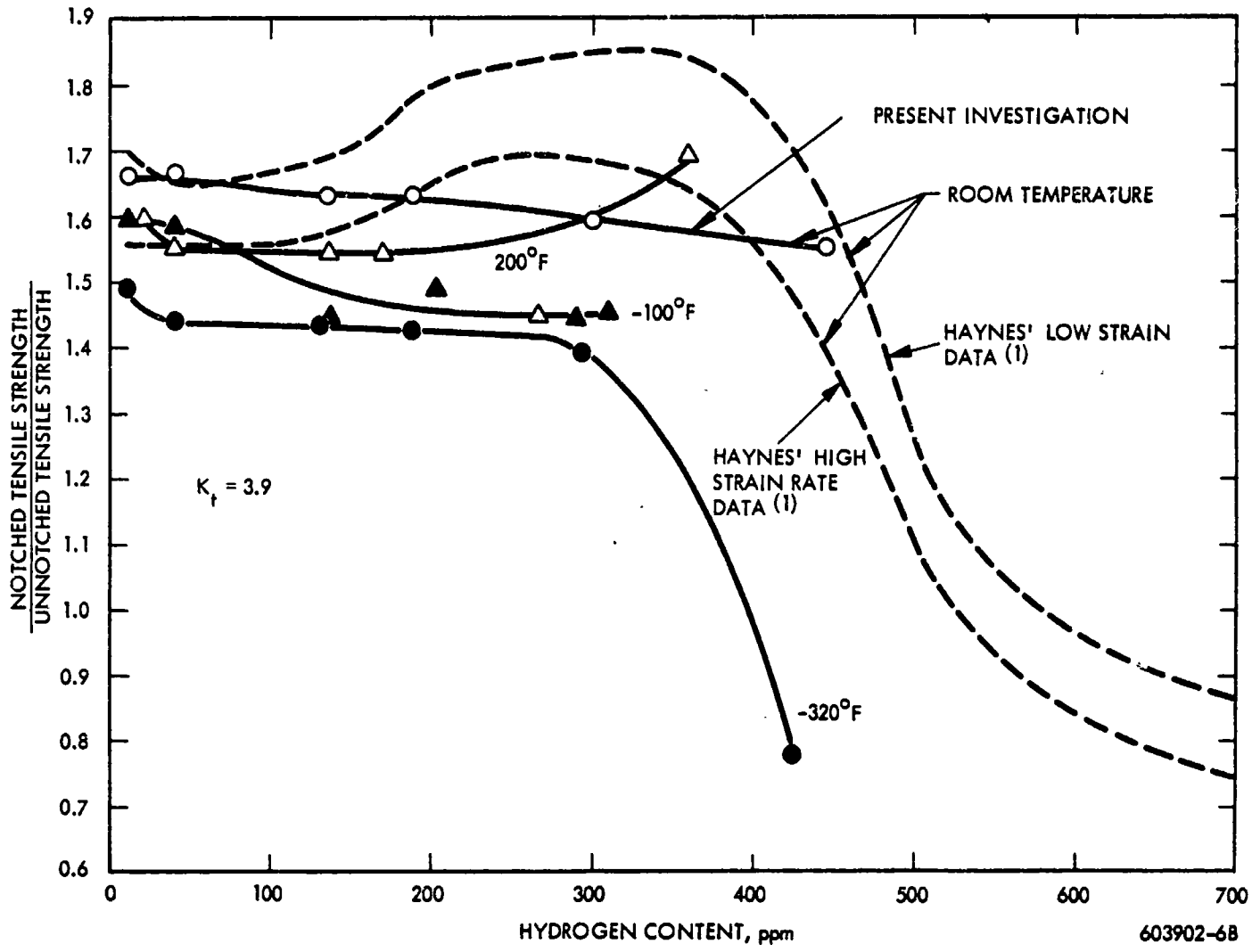


FIGURE 6 - The Effect of Hydrogen on the Notched : Unnotched Tensile Strength Ratio of Ti-5Al-2.5Sn

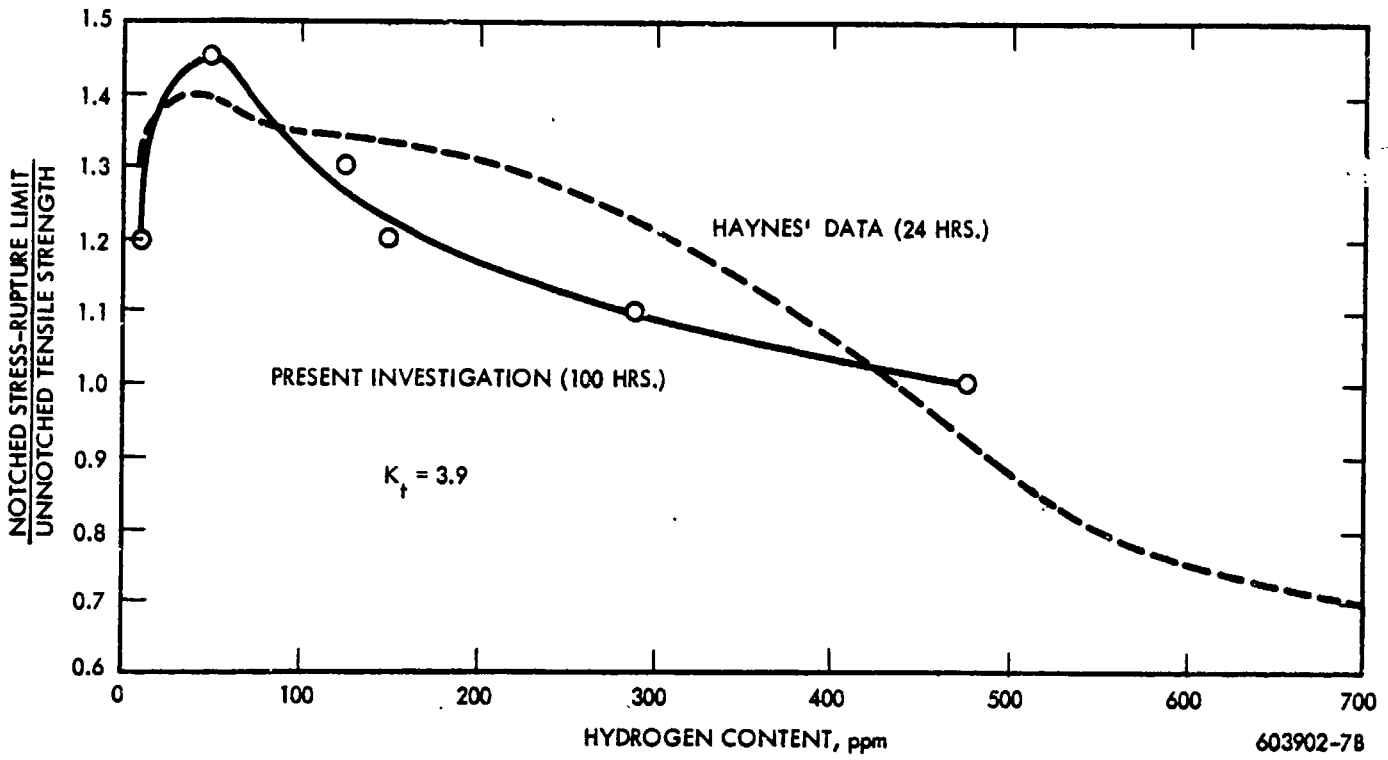


FIGURE 7 - The Effect of Hydrogen on the Room-Temperature Notched Stress-Rupture Limit of Ti-5Al-2.5Sn

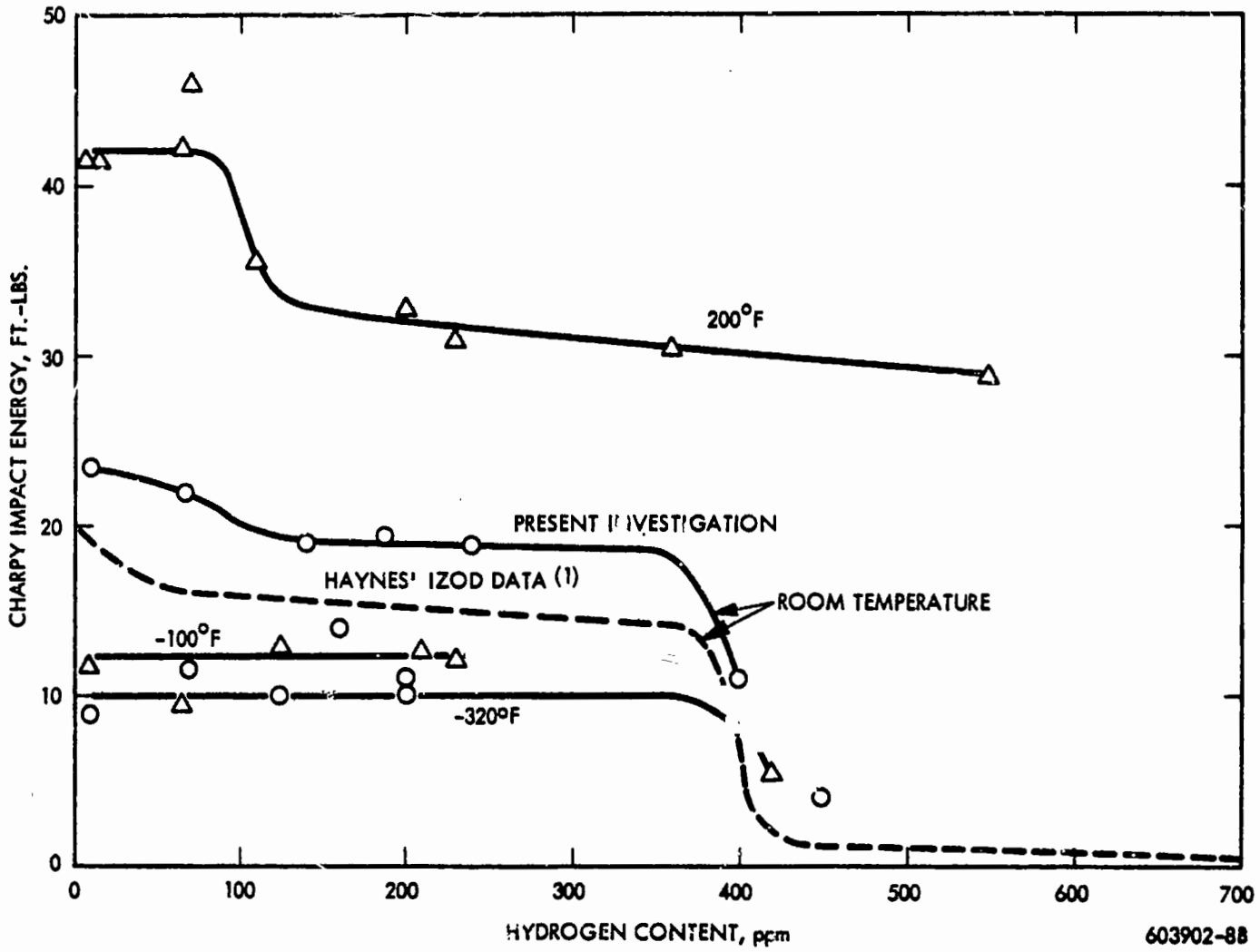
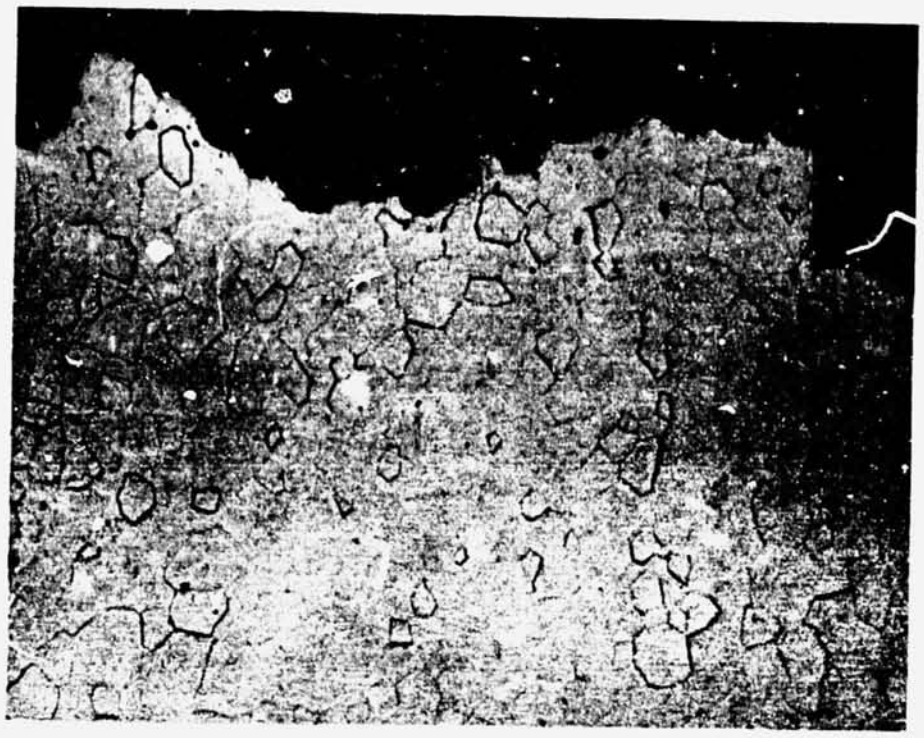


FIGURE 8 - The Effect of Hydrogen on the Impact Properties of Ti-5Al-2.5Sn



Etched

200x

(a) 6 ppm Hydrogen

Etched

200x

(b) 153 ppm Hydrogen

Figure 9 Microstructures of Notched Stress-Rupture Specimens

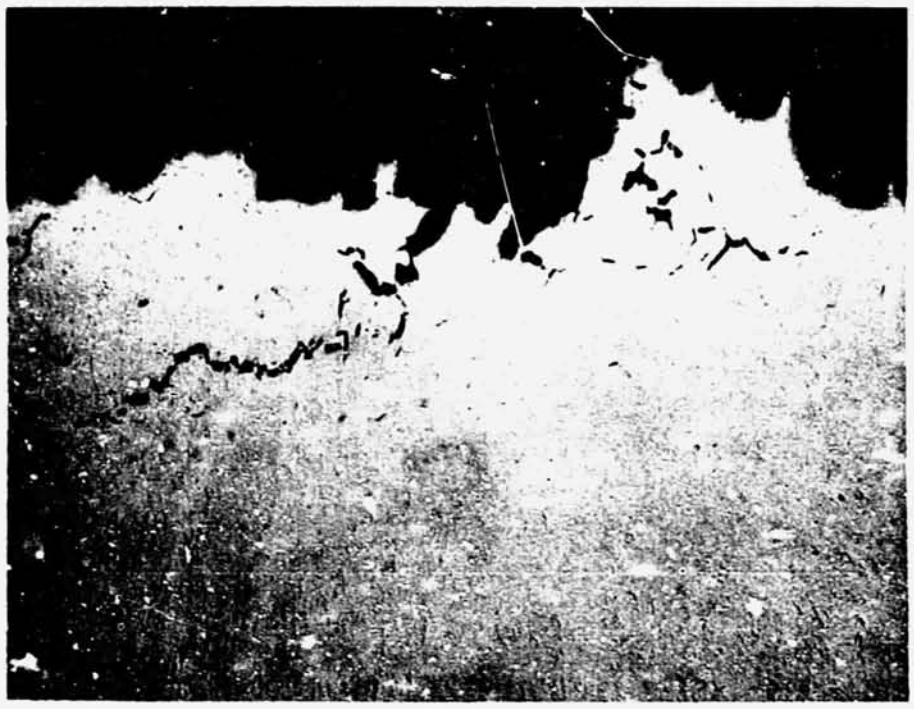
Etched 200x

(c) 288 ppm Hydrogen

Etched 200x

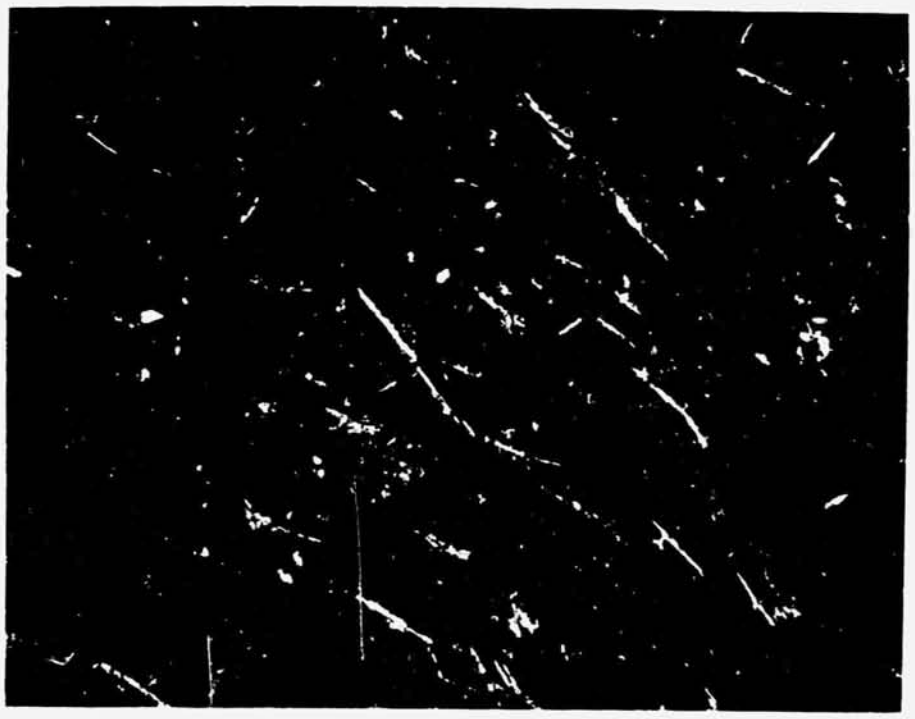
(d) 473 ppm Hydrogen

Figure 9 (Continued)



Unetched 200x

(e) 473 ppm Hydrogen



Etched 500x

Polarized Light

(f) 473 ppm Hydrogen

Figure 9 (Concluded)