

# Effects of Hydrogen on 5Al-2.5Sn ELI Titanium

W. E. HENSLEY\* AND W. T. CHANDLER†

*Rockwell, A Division of North American Rockwell Corporation, Canoga Park, Calif.*

A program was conducted to determine the effects of hydrogen on 5Al-2.5Sn ELI titanium alloy under selected conditions. Tensile tests were conducted at  $-423^{\circ}\text{F}$  on specimens abraded in hydrogen, helium, and air, stressed, and temperature-cycled between  $400^{\circ}$  or  $800^{\circ}\text{F}$  and  $-423^{\circ}\text{F}$  prior to testing. In all cases, there was no evidence of embrittlement as determined by tensile tests. Under simulated fretting conditions in gaseous hydrogen, a powder was formed that was identified as  $\text{TiH}_2$ . Abrasion of titanium in flowing hydrogen at temperatures of  $400^{\circ}$  and  $800^{\circ}\text{F}$  formed  $\text{TiH}_2$ , and the external temperature was high enough to form the hydride phase in the alloy.

## Introduction

THE 5Al-2.5Sn ELI titanium alloy is being considered for use in components (e.g., liquid hydrogen pump components and storage vessels), which are highly stressed while in contact with liquid hydrogen. During operation, the titanium alloy will, on occasion, warm up so that it is in con-

tact with gaseous hydrogen at temperatures above room temperatures. Also, for rotating components, it will, on occasion, rub against other alloys while in hydrogen. Prior workers<sup>1</sup> have found that it is possible to form  $\text{TiH}_2$  by rubbing titanium against iron and nickel in hydrogen. Also, thread fretting of titanium alloy against the same alloy in hydrogen has led to extensive  $\text{TiH}_2$  powder formation. The titanium-hydrogen constitution diagram is shown in Fig. 1.

The program discussed herein was conducted to determine the effect of various conditions on the properties of the 5Al-2.5Sn titanium alloy at liquid hydrogen temperatures and on the tendency of this alloy to form hydride. The tensile specimens and the specimens for the rubbing tests were made from  $\frac{5}{16}$ -in. bar stock. Vendor-certified (Titanium Metals Corporation of America) chemical analysis and mechanical properties are reported in Table 1. The alloy was used in the as-received condition with no special heat treatment or preparation. Elevated temperature abrasion tests were conducted on 0.125-in.-thick sheets of unknown history.

## Effects of Abrasion, Stressing, and Temperature Cycling on Tensile Properties at $-423^{\circ}\text{F}$

Tensile tests were conducted at  $-423^{\circ}\text{F}$  on specimens that had been abraded, stressed, and temperature-cycled between  $400^{\circ}$  or  $800^{\circ}\text{F}$  and  $-423^{\circ}\text{F}$  in hydrogen prior to tensile testing. Specimens were stressed at 35 ksi after the abrading and prior to temperature cycling. During the temperature cycling, the specimens were held for 30 min at both the maximum temperature and  $-423^{\circ}\text{F}$ . Similar tests were performed in air and helium for comparison. The novel apparatus depicted in Figs. 2 and 3 was used in these tests. The specimens were 5 in. long with a 1-in.-long reduced section of 0.125 in. diam and with threaded ends for gripping. The abrader was forced against the surface of the specimen by a spring, and a flywheel was placed around the specimen, which held the abrading tool in place. The specimen, with the abrading device assembled around it, was sealed into a container with a bellows at one end, which provided sufficient extension so that the specimen could be tested to failure. It was felt that sliding seals would not provide sufficiently reli-

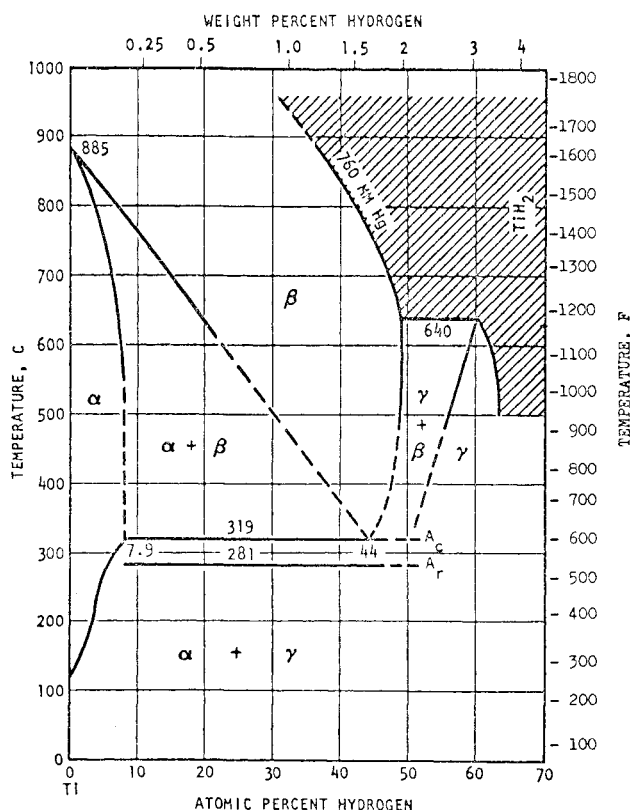


Fig. 1 Titanium-hydrogen constitution diagram.

Presented as Paper 69-585 at the AIAA 5th Propulsion Joint Specialist Conference, U.S. Air Force Academy, Colo., June 9-13, 1969; submitted June 18, 1969; revision received September 2, 1969. The authors gratefully acknowledge the financial support of NASA under Contract NASS-19. The authors are indebted to the following for their contributions to these investigations: G. E. Dyer, H. G. Hayes, J. N. Lamb, T. Schroeder, and J. Testa.

\* Member of the Technical Staff, Processing Metallurgy, Research Division. Member AIAA.

† Principal Scientist, Physical and Mechanical Metallurgy, Research Division. Member AIAA.

Table 1 Chemical analysis (% by weight) and mechanical properties of 5Al-2.5Sn ELI Ti as received

C	Fe	N <sub>2</sub>	Al	H <sub>2</sub>	O <sub>2</sub>	Mn	Sn
0.011	0.09	0.006	5.47	0.0122	0.09	0.02	2.70
$\sigma_{ty}$ , ksi		$\sigma_{tu}$ , ksi		$\Delta l$ , %		$\Delta A$ , %	
117.0		121.5		12.5		43.0	

**Table 2 Results of tensile tests of 5Al-2.5Sn ELI Ti at  $-423^{\circ}\text{F}$  (specimens temperature cycled between  $400^{\circ}$  and  $-423^{\circ}\text{F}$  four times prior to test)**

Abrader	Number of tests	$\sigma_{tu}$ , ksi, lowest avg.	$\Delta A$ , %, lowest avg.
Control, not temperature-cycled or abraded		214	30.2
A) <sup>a</sup> None	8	211	25.2
440 C	3	208	24.8
Inco 718	6	184	31.4
Fe File	3	200	34.7
Ti	6	169	30.9
A-286	3	198	34.2
Tens-50	3	165	32.5
B) <sup>b</sup> None	2	200	38.0
Inco 718	2	190	36.0
Ti	2	190	40.0
440 C	2	180	43.0
C) <sup>c</sup> None		198	38.3
Ti	2	188	38.8
440 C	2	185	36.2
Inco 718	2	184	38.0
D) <sup>d</sup> None	4	199	37.1
440 C	3	190	35.0
440 C	3	175	37.6

<sup>a</sup> Conducted in  $\text{H}_2$  after cycling four times between  $400^{\circ}$  and  $-423^{\circ}\text{F}$ .

<sup>b</sup> In  $\text{H}_2$ , after four cycles, between  $800^{\circ}$  and  $-423^{\circ}\text{F}$ .

<sup>c</sup> In  $\text{H}_2$ , after ten cycles, between  $800^{\circ}$  and  $-423^{\circ}\text{F}$ .

<sup>d</sup> In  $\text{He}$  or air, after four cycles, between  $400^{\circ}$  and  $-423^{\circ}\text{F}$ .

able hydrogen sealing through the temperature cycling procedure.

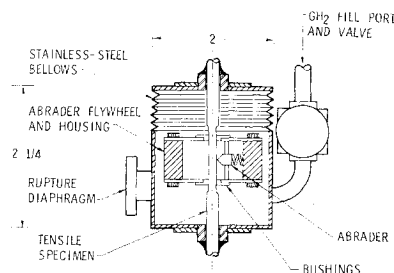
Assembly of the specimen into the container consisted of silver brazing an end plate on one end of the specimen, placing the abrading device around the specimen, inserting the specimen and abrading device in the container, welding the end plates on the container, and silver brazing the specimen to the end plate at the bellows end of the container. The container was then evacuated to  $10^{-4}$  torr and backfilled with high-purity hydrogen (1 ppm total impurities) four times. After the last hydrogen fill, the valve was closed.

The specimen was then abraded. The specimen and container assembly was spun in a lathe. The Teflon bushings in the abrader and plates gripped the specimen sufficiently so that the flywheel would finally reach essentially the speed of the specimen. The lathe (and specimen) was stopped suddenly and the inertia of the flywheel kept the abrader spinning around the specimen, and the abrading tool, as designed, "dug in" and abraded the specimen.

After abrading, the specimen was stressed to 35 ksi in the stress frame shown in Fig. 3. Strain gages attached to the loading columns accurately determined the load. The entire assembly was then temperature cycled 4 or 10 times. Then the stress frames were removed and the specimens were loaded into the tensile machine. The specimen and container were immersed in liquid hydrogen, the diaphragm shown in Fig. 2 was broken, allowing  $\text{LH}_2$  to flow into the container, and a tensile test was conducted with the specimen immersed in the  $\text{LH}_2$ . The ultimate tensile strength  $\sigma_{tu}$  and reduction of area  $\Delta A$  of the specimen were determined. Post-test examination of the failed specimen included electron fractography of the fracture surface, vacuum fusion analysis for hydrogen, and light microscopy to determine the effect of the hydrogen environment on the microstructure and, more particularly, to look for hydride formation.

Part A of Table 2 presents the results of abrading, stressing, and temperature cycling four times between  $400^{\circ}$  and  $-423^{\circ}\text{F}$  prior to tensile testing. In general, the tensile strengths of abraded specimens were lower while the ductilities, as measured by reduction of area, were higher than for unabraded specimens. Part B of Table 2 presents the results

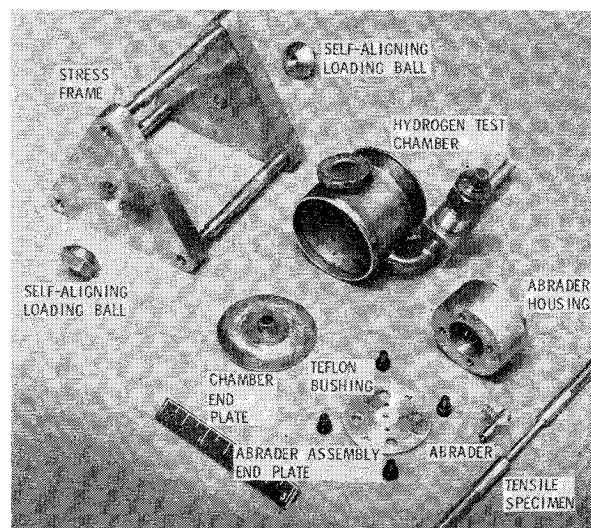
**Fig. 2 Schematic of apparatus for abrading and tensile testing 5Al-2.5Sn ELI Ti specimens in hydrogen.**



of temperature cycling four times between  $800^{\circ}$  and  $-423^{\circ}\text{F}$ . Again, the strength was lower for abraded specimens while the reduction of area was higher. Part C presents data for temperature cycling 10 times between  $800^{\circ}$  and  $-423^{\circ}\text{F}$ . The effects of abrading in helium and in air prior to temperature cycling between  $400^{\circ}$  and  $-423^{\circ}\text{F}$  are found in part D. A comparison of the test results presented in Table 2 shows that the greatest average decrease in strength after cycling between  $400^{\circ}$  and  $-423^{\circ}\text{F}$  occurred after abrading with Tens-50 aluminum, a decrease of 14.2%. Abrading with titanium gave a 6.4% decrease while abrading with A-286 gave a 7.2% decrease. The other decreases ranged from 0 to 5.5%. Abrading with 440 C, an iron file, or Inco 718 had little or no effect on the tensile properties.

A comparison of the lowest strength in each series is believed to be more significant, however, because it was possible that the abrader may not have actually effectively abraded a particular specimen to produce a fresh oxygen free surface for reaction with hydrogen. Using the lowest strength criteria, abrading with titanium, Inco 718, and Tens-50 aluminum caused decreases in strength of 20, 13, and 22%, respectively. Abrading with 440 C, A-286, and iron files decreased the minimum strength 1, 6, and 5%, respectively. The reductions of area were increased by the same order of magnitude for all abrading materials except 440 C, which showed little change. The average change in reduction of area ranged from 13 to 19%, while the minimum change ranged from 24 to 35% for all materials except 440 C.

Unexpectedly, abrading with 440 C showed the greatest decrease both in average and minimum strengths (12.2 and 10.7%). Abrading with Inco 718 resulted in a 9.7% average and 5.8% minimum decrease in strength, while abrading with titanium resulted in a 5.0% average and 5.8% minimum decrease in strength. The increase in reduction of area was not as great after the  $800^{\circ}$  to  $-423^{\circ}\text{F}$  temperature cycle as with the  $400^{\circ}$  to  $-423^{\circ}\text{F}$  cycle, nor was it as consistent.



**Fig. 3 Components of test apparatus for abrading and tensile testing 5Al-2.5Sn ELI Ti specimens in hydrogen.**

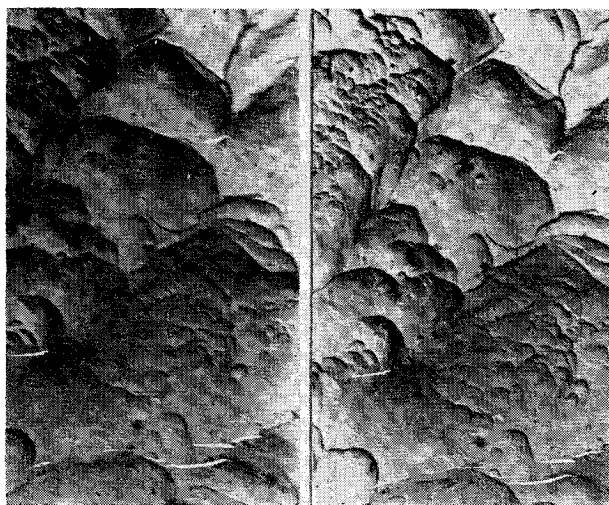


Fig. 4 Electron fractograph showing typical ductile (dimple) failure (stereo pairs).

The average increases were 19.4% for 440 C, 4.0% for titanium, and 2.1% for Inco 718. The minimum increases were 0.5% for titanium, 7.9% for Inco 718, and 0.9% for 440 C. Abrasion with 440 C showed the greatest change in both reduction of area and strength.

Temperature cycling between 400° and -423°F without abrading produced a 3.2% decrease in average tensile strength compared to a specimen that had not been temperature cycled. Specimens abraded with 440 C showed a 15.6% decrease in strength between temperature cycling between 800° and -423°F and temperature cycling between 400° and -423°F. Specimens abraded with Inco 718 had only an 8.2% decrease and with titanium only a 6.8% decrease between temperature cycling between 800° and -423°F and 400° and -423°F. These results clearly show that increasing the maximum cycling temperature produced a more severe effect on strength, and that abrading plus increasing the maximum cycling temperature decreased the strength even more.

During the program, the question arose as to whether filling the container once with hydrogen was sufficient to affect the material: Was the hydrogen used up before the full effects took place? To answer this question, a second procedure was initiated. The procedure of filling the can with hydrogen and abrading the specimen was carried out ten times prior to temperature cycling between 800° and -423°F. The average differences between the two abrasion treatments were found to be 0.3 to 1.5%, which were considered insignificant. These comparisons show clearly that the effects that occurred did so with only one hydrogen filling of the cans and that more than one filling was not necessary.

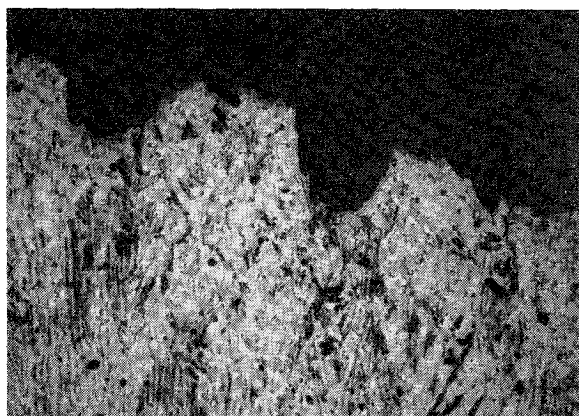


Fig. 5 Fracture edge typical of all tensile-tested specimens regardless of prior maximum temperature.

Table 3 Vacuum fusion analyses of tensile specimens for hydrogen content

Abrader	Liquid	Hydrogen, ppm
None	H <sub>2</sub>	143, 178
Inco 718	H <sub>2</sub>	144, 175
Ti	H <sub>2</sub>	123, 140
440 C	H <sub>2</sub>	170, 174
Tens-50	H <sub>2</sub>	200
Tens-50	He	145
None	He	162
440 C	He	145
440 C	Air	160

For comparison, it was decided to test abrasion effects in other environments (liquid air and helium) with 440 C as the abrading material. Unexpectedly, these tests showed that there was a decrease in strength due to testing in He and due to abrading in He or air. Compared to testing in LH<sub>2</sub> without abrasion, testing in He showed a 5.5% decrease in both the average and minimum strengths. Abrading and testing in He resulted in a 17.9 and 17.0% decrease in strength. All tests in He and air were conducted at -423°F. The He or air pressure was approximately 0.07 atm at -423°F.

Electron microscope fractographic analyses were conducted on representative specimens to determine the mode of failure and the effect of hydrogen. In all cases, regardless of the abrader material and regardless of the environment, the fracture mode was ductile (dimpled appearance). In no case was there any evidence of a brittle fracture. The dimpled structure shown in Fig. 4 is typical of all the specimens examined.

Filed chips taken from the fracture surfaces of representative tensile specimen were subjected to vacuum fusion analyses for LH<sub>2</sub>. These analyses showed no increase or decrease in LH<sub>2</sub> due to abrading in LH<sub>2</sub>. In fact, the analyses of the specimens abraded in He and air showed as much LH<sub>2</sub> as those abraded in LH<sub>2</sub> (Table 3).

Optical metallographic examinations were made of the fracture ends of tensile specimens. To show the presence of TiH<sub>2</sub>, these samples were polished perpendicular to the fracture surface and etched with 30 parts lactic acid, 30 parts nitric acid, and 1 part hydrofluoric acid and viewed under polarized light. Under these conditions, TiH<sub>2</sub> is found to have bright contrast. None of the fracture areas showed any TiH<sub>2</sub> in the microstructure. Microscopic examination of the fracture surfaces indicated that all failures were ductile by evidence of plastic deformation at the fracture surface. Figure 5 represents a typical fracture edge. Figure 6 shows a typical microstructure in the unaffected area.

In summary, abrading and temperature cycling prior to tensile testing at -423°F did not embrittle the alloy. A loss of tensile strength occurred, but this was accompanied by a gain in ductility. There was no evidence of TiH<sub>2</sub> formation or diffusion of hydrogen into the titanium matrix resulting from the tests performed. Prior investigations have shown embrittlement and loss of impact strength at room temperature with as little as 150 ppm H<sub>2</sub> due to hydride formation. However, specimens in this program contained 170 ppm H<sub>2</sub> with no embrittlement. No prior work conducted on Ti can account for the increase in ductility or loss in strength found in this program. However, the changes

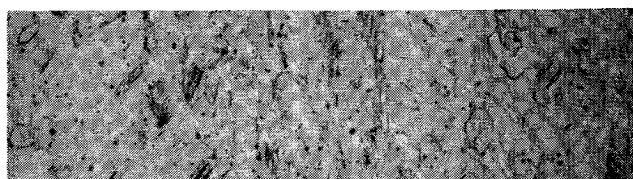


Fig. 6 Center of typical specimen.

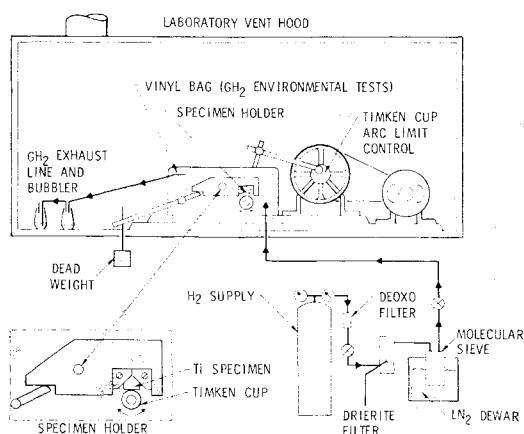
**Table 4 Results from abrading 5Al-2.5Sn ELI Ti specimens against one another in hydrogen at room temperature in a MacMillan tester**

Test No.	Load on arm, lb	Arc of trace, deg	Environment	Number of cycles	Remarks
1	10	22½	H <sub>2</sub>	58	Metal squeak from start; gray powder formed; metal smeared ¾ in. wide; bag ignited.
2	5	22½	H <sub>2</sub>	108	Metal squeak from start; not as much powder; metal smeared ¾ in. wide.
3	10	10	H <sub>2</sub>	104	Metal squeak from start; similar to test No. 1; smeared area ¼ in. wide.
4	10	10	Air	103	No squeak until 40 cycles; smeared area ⅙ inch wide; small amount of powder after 103 cycles; less powder than in test No. 3.
5	10	10	H <sub>2</sub>	397	Spark at 204 cycles; ignition at 397 cycles; heavy wear and large amount of powder.
7	10	10	H <sub>2</sub>	2500	Spark at 415 cycles; spark at 744 cycles; spark at 910 cycles; spark at 1082 cycles; test stopped and bag flushed with H <sub>2</sub> after each spark; Ti specimen heavily scarred; large amount of powder.
8	10	10	Air	2500	Started quiet; squeak again at 50 cycles; smaller amount of powder than in test No. 7.

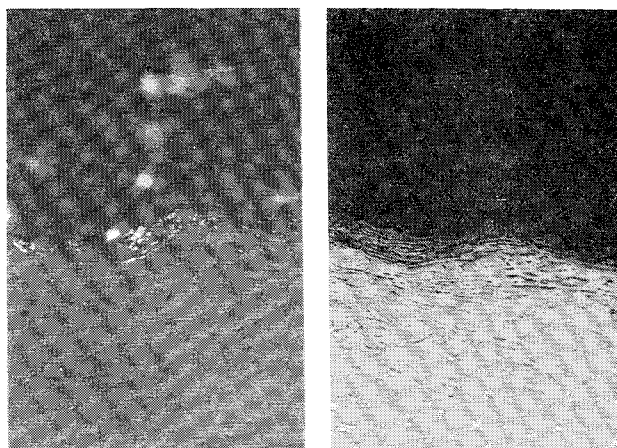
appear to be real because they are too large to be attributed to data scatter.

### Effect of Rubbing the Ti Alloy against Itself in H<sub>2</sub> at Ambient Temperature

A MacMillan tester, which is used for bearing friction tests, was adapted to simulate vibratory rubbing of the Ti alloy against itself in an H<sub>2</sub> atmosphere. The experimental setup is shown in the schematic drawing in Fig. 7. In this test, a 5Al-2.5Sn ELI titanium Timken cup was vibrated against a specimen made of the same material. The specimen was forced against the cup by a deadweight load applied through a level. The Timken cup oscillated through an arc of 22.5° or 10° to provide the simulated vibratory motion desired. The frequency of oscillation was ~2.8 Hz. A portion of the apparatus and test specimens were contained in a plastic bag. Hydrogen was flowed through the plastic bag from a tank of high-purity hydrogen that was further purified to less than 3-ppm impurities. The GH<sub>2</sub> exhausted through a



**Fig. 7 MacMillan tester.**



**Fig. 8 Fretted surface of specimen tested on MacMillan tester shows TiH<sub>2</sub> at surface.**

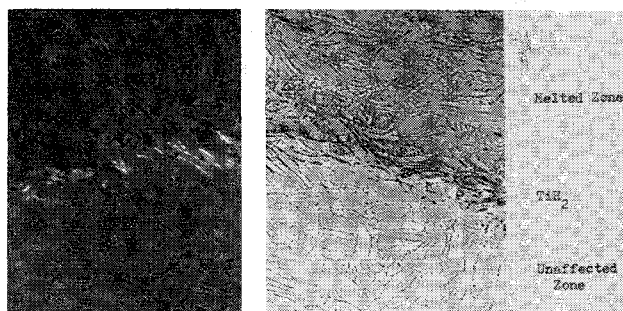
bubbler system. Tests were conducted at ambient temperature. The whole apparatus was placed in a laboratory hood for test.

Analysis of the test results consisted of x-ray diffraction and vacuum fusion analysis of the powder formed. Additional metallographic studies were performed to identify changes in microstructure of the specimen due to testing. Table 4 summarizes the tests performed. Tests conducted under the higher load (i.e., 10 lb) and oscillated through the greater arc (22.5°) produced more wear and much more powder. Abrading in H<sub>2</sub> caused a dark powder to form, which was identified by x-ray diffraction analysis as TiH<sub>2</sub>. In addition, gas analyses conducted on the powders showed 3.27% H<sub>2</sub> by weight in the powder. This calculated out to an H<sub>2</sub> to Ti atom fraction of 1.6, which is in good agreement with the stoichiometric TiH<sub>2</sub>. Tests conducted in air produced a gray powder, which was identified as TiN and TiO<sub>2</sub> by x-ray diffraction.

Optical microscope examination of abraded specimens clearly showed the presence of TiH<sub>2</sub> at the abraded surface (Figs. 8 and 9). It is of interest that in one area where melting occurred, the TiH<sub>2</sub> was only found at the interface between the cast structure and the unaffected structure.

No particularly noticeable noise was heard during testing in air until after ~50 Hz. On the other hand, a definite "squeak" was heard from the initial cycle in the H<sub>2</sub> tests, which indicates that the oxide film formed during the air tests provided a film lubricant and less friction. It also indicates that the oxide film was removed immediately in H<sub>2</sub>.

An unexpected phenomenon was observed during the hydrogen tests. Although no detectable leaks could be found in the plastic bag, sparks were observed to jump from the outside of the plastic bag to the test frame. This caused the plastic bags to burn in several instances. Every precaution had been taken to insure that the test apparatus was grounded; therefore, the sparks are not believed to be due



**Fig. 9 Fretted surface of specimen tested on MacMillan tester shows melted loops.**

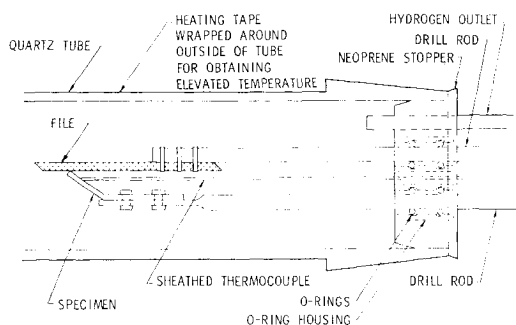


Fig. 10 Apparatus for abrading in hydrogen at elevated temperatures.

to static electricity. It was found that by flowing nitrogen over the outside of the bag, the frequency of the sparking could be cut down. Also, it was noted that stopping the test and flushing the system with hydrogen when sparks were observed minimized the sparking. It was not within the scope of this program to investigate the phenomenon further, but it is believed that further investigations are warranted with other materials in other environments.

This phase of the investigation was initiated to simulate fretting between threaded components and to determine whether fretting at ambient temperatures could produce  $\text{TiH}_2$ . Tests conducted did indeed produce  $\text{TiH}_2$  at ambient bulk temperature in flowing  $\text{H}_2$ . An explanation for this phenomenon follows. The fretting action continually supplied a fresh oxide-free surface for the  $\text{H}_2$  gas. Heat generated by friction raised the surface temperature high enough, probably near the melting point, for the  $\text{TiH}_2$  reaction to take place. At these temperatures, the  $\text{H}_2$  would be absorbed very rapidly. As the temperature was lowered,  $\text{TiH}_2$  was formed.

The 12.7% increase in volume when  $\text{Ti} + \text{H}_2 \rightarrow \text{TiH}_2$  and the brittleness of the hydride causes spalling and flaking to take place. The investigation conducted on the MacMillan tester clearly demonstrated that  $\text{TiH}_2$  can form in  $\text{H}_2$  at ambient temperatures due to the fretting of two mating surfaces, and points to the need for design care where threaded components are subject to fretting in  $\text{H}_2$ .

### Effect of Filing 5 Al-2.5 Sn ELI Titanium in Hydrogen at Elevated Temperatures

This experiment was performed to determine whether or not  $\text{TiH}_2$  could be formed by filing the 5Al-2.5Sn ELI titanium alloy in hydrogen at 400° and 800°F. The apparatus used for abrading specimens in flowing  $\text{H}_2$  is shown schematically in Fig. 10. A 0.070-in.-thick specimen was abraded with an iron file at the desired temperature in a quartz tube. Hydrogen was flowed through the tube at 1 atm pressure. The  $\text{H}_2$  used in this experiment was purified to less than 3-

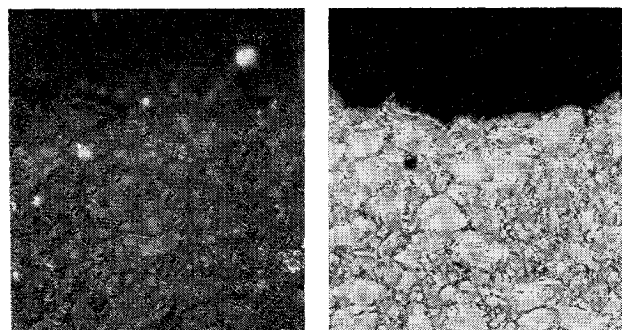


Fig. 11 Specimen abraded in flowing hydrogen at 400°F, showing hydrided area at edge (300×).

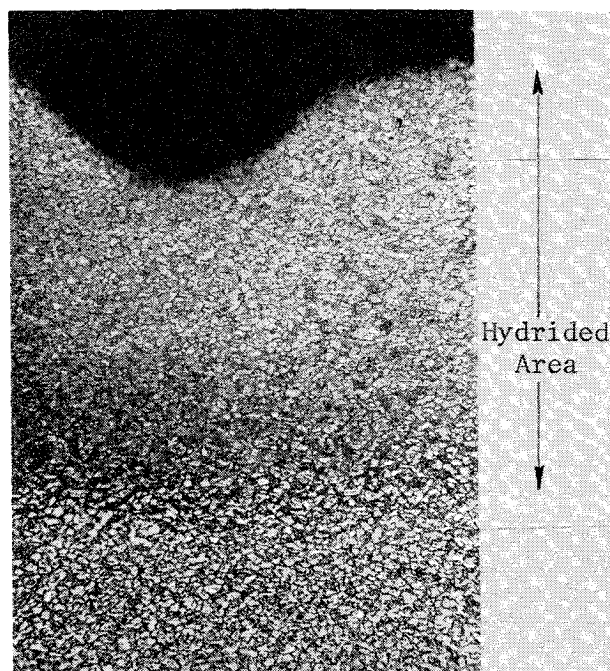


Fig. 12 Specimen abraded in flowing hydrogen at 800°F, showing  $\text{TiH}_2$  formation (75×).

ppm total impurities. The specimen was brought to temperature by means of a heating tape surrounding the tube. The specimen temperature was measured by a thermocouple attached to the specimen. After the tube was brought to temperature, the specimen was abraded. Following abrasion, the specimen was cooled in  $\text{H}_2$  at  $\sim 5.5^\circ\text{F}/\text{min}$ . After reaching room temperature, the specimen was examined microscopically for changes in microstructure due to testing.

Abrading in  $\text{H}_2$  formed  $\text{TiH}_2$  at both 400° and 800°F. The hydride was detected by microscopic examination. Specimens were polished and etched so that the hydride contrasted with the matrix under polarized light. Examples of the hydride structures may be seen in Figs. 11-13. A different microstructure was formed by abrading at the two temperatures. The hydride phase formed at 400°F etched darker at the surface and contrasted with the matrix at the surface. The hydride that formed at 800°F had two distinct areas. The area away from the edge etched differently and contrasted with the matrix under polarized light, but the area next to the edge showed no contrast. As may be seen from the phase diagram shown in Fig. 1, 400°F is below the eutectoid temperature. This, apparently, caused the formation of the platelike structure, which contrasts with the matrix. The 800°F treatment was above the eutectoid temperature. The structure that formed had a different morphology and composition at the surface. The hydride structure formed at the grain boundaries did not contrast under polar-

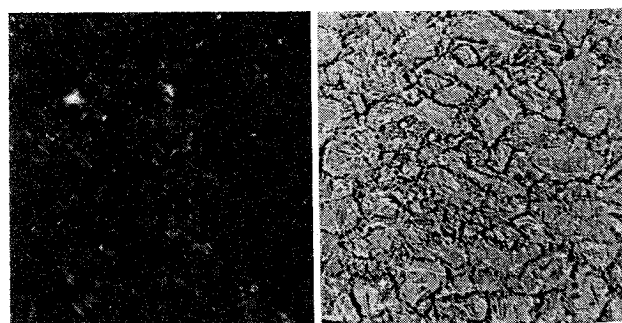


Fig. 13 Specimen abraded in hydrogen at 800°F, showing  $\text{TiH}_2$  in dark band away from edge (300×).



ized light. Away from the edge, however, the temperature was lower and the hydride that formed had the same structure as that found at 400°F. This experiment clearly demonstrated that  $\text{TiH}_2$  will form when the 5Al-2.5Sn ELI titanium alloy is abrading in flowing  $\text{H}_2$  at either 400° or 800°F.

It is believed that the abrading removed the protective oxide film, which exposed a fresh surface to the  $\text{H}_2$ . The 400° or 800°F temperature was high enough so that the reaction  $\text{Ti} + \text{H}_2 \rightarrow \text{TiH}_2$  could occur.

It has been demonstrated by other researchers<sup>2-4</sup> that  $\text{TiH}_2$  causes brittleness and spalling. The heat required to produce temperatures high enough for  $\text{H}_2$  to react with Ti can come from fretting as was shown by rubbing titanium against itself in the previous section of this investigation. In either case, the hydride can form.

### Conclusions

1) No hydrogen embrittlement was detected during tests performed in this investigation.

2) Losses of strength and increase in ductility at -423°F were found only after abrading. Losses in strength of 13-21% resulted from abrading with titanium, Inco 718, and Tens-50 aluminum; increases in ductility were 17-19%. The decrease in strength and increase in ductility appear to

be too large to be attributed to data scatter; therefore, the changes are considered significant.

3)  $\text{TiH}_2$  can be formed when the 5Al-2.5Sn ELI Ti alloy is rubbed against itself in  $\text{H}_2$  at ambient temperatures.

4) Sparks were generated outside of the plastic container during testing in hydrogen. The sources of these sparks are unknown. Previous tests in another program using nitrogen gas testing dry film lubricants did not produce these sparks. An investigation should be conducted to determine the nature and cause of the sparking, because of its possible deleterious effects to  $\text{H}_2$  systems.

### References

<sup>1</sup> McQuillan, A. D., "The Effect of the Elements of the First Long Period of the  $\alpha \rightleftharpoons \beta$  Transformation in Titanium," *Journal of the Institute of Metals*, Vol. 80, March 1952, pp. 363 and 368.

<sup>2</sup> Koehl, B. G. et al., "Continuation of the Investigation of the Reaction of Titanium With Hydrogen," Contract NAS9-5298, May 1967, Battelle Memorial Institute, Columbus, Ohio.

<sup>3</sup> Halchak, J. A., personal communication, Sept. 1967, Rocketdyne, Canoga Park, Calif.

<sup>4</sup> Phillips, A., Kerlins, V., and Whiteson, B. V., "Electron Fractography Handbook," TR ML-TDR-64-416, Jan. 1965, Air Force Materials Lab., Wright-Patterson Air Force Base, Ohio.