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FAILURE ANALYSIS OF TX - 15a TX GETTER WIRE USED FOR SUBLIMATION
IN THE VACUUM CHAMBERS OF THE TANDEM MIRROR EXPERIMENT

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FAILURE ANALYSIS OF Ti-15% TA GETTER WIRE USED FOR SUBLIMATION IN THE VACUUM CHAMBERS OF THE TANDEM MIRROR EXPERIMENT

R. P. Kershaw, R. J. Gross, and E. N. C. Dalder

The Tandem Mirror Experiment uses Ti-15% Ta getter wire for sublimation in the vacuum chambers in which the magnets are located. These wires have failed prematurely in service, resulting in increased costs and downtime.

We have used optical metallography to show that the reason for these failures was the cycling of the material through the alpha-beta transition temperature, causing alpha-titanium precipitation at the grain boundaries, depression of the melting temperatures of those boundaries, and the subsequent melting of those boundaries in areas where the wires had achieved localized higher temperatures.

INTRODUCTION

Titanium vapor deposited on the surface of a vacuum chamber removes some gases by chemisorption. This method is selective in that it only removes hydrogen, oxygen, nitrogen, water vapor, carbon monoxide, and carbon dioxide.

Titanium is alloyed with tantalum to stabilize the beta phase to room temperature, thus strengthening the material and raising the melting point of the material.

The operational life of these wires is a function of sublimation rates (gm/hr), the number of cycles through the alpha-beta transition temperature, and pressure during sublimation. Sublimation rates that result in a higher than needed sublimation of titanium reduce filament life. Higher pressures decrease pumping speed (Fig. 1). These pressures are dependent on the rate of titanium sublimation. Titanium-limited pumping results when the getterable gases combine with the titanium as fast as the latter is deposited.¹ This means that continuous gettering is required.

FAILURE ANALYSIS

Examination of two wires received from the supplier (Teledyne Wah Chang-Albany), that showed different degrees of ductility, revealed different microstructures. The microstructure of the less ductile wire is that of a cold-drawn material (Fig. 2). The grains are elongated in the direction of drawing and are heavily deformed, while the more ductile wire has a microstructure that exhibits recovery, i.e. the first stage in the removal of work-hardening effects (Fig. 3). The recovery occurred without the motion of grain boundaries.

Two wires that had performed successfully in TMX-U were also examined. It is evident that, although both wires have an acicular alpha in a matrix of beta

structure and have large grains, there is a large difference in the coarseness of the microstructure, one being much finer than the other. This difference in microstructure indicates that the two wires were not cooled at the same rate. Slower cooling rates result in coarser structures (Figs. 4 and 5). Both photomicrographs exhibit large grain size. The wire with the coarse acicular structure shows evidence of voids or hydrogen porosity (Fig. 6).

Several specimens from wires that broke in service show evidence of the cycling through the alpha-beta transition temperature during operation. In Fig. 7, note that the alpha phase (light) precipitated along the grain boundaries and within the grains (light rosettes surrounded by dark beta). Further investigation showed that the precipitated alpha had melted (Figs. 8-10).

DISCUSSION AND CONCLUSION

Hydrogen is not as soluble in the alpha phase as it is in the beta phase.² Upon cooling from the operating temperature range, which is above the alpha-beta transformation temperature (transus) of about 840 °C for a Ti-15% Ta alloy, any large amount of hydrogen (greater than 1840 wppm)* that cannot diffuse out of the alpha phase between 840 °C and 319 °C, will form gas pockets, called hydrogen porosity, in the alpha phase. Below 319 °C, a phase called gamma forms within the alpha phase; this is the hydride phase. This titanium hydride phase precipitates in the alpha as titanium hydride needles, is very brittle,³ and will crack, due to the thermal stresses built up by repeated cycling between idling or room temperature and operating temperature.

Hydrogen porosity and/or cracking, due to the presence of the brittle titanium hydride phase, lowers the cross-sectional area of the getter wire and results in local areas with higher electrical resistance and therefore local higher temperatures.

Examination of the titanium-tantalum phase diagram (Fig. 11) shows that the solubility of tantalum in alpha titanium (about 13% at room temperature) is much lower than in beta titanium (where tantalum and titanium form a continuous series of solid solutions). Every time the getter wire is heated to the operating temperature of about 1600 to 1750 °C, the following series of events takes place:

- At 840 °C, the transformation of alpha titanium to beta titanium is complete.
- Starting at approximately 920 °C, the grain size of the beta-titanium increases greatly.³
- At 1600 °C, hydrogen and other getterable gases react with the sublimated titanium and deposit on the chamber walls.
- Throughout the temperature range, hydrogen is absorbed into the getter wire.

Every time the getter wire is cooled rapidly from 1750 to 1600 °C down to room temperature, the following sequence of events takes place:

*wppm = weight percent per million

- At 1600 to 840 °C, hydrogen is still being picked up by the getter wire, until the beta phase is saturated with hydrogen in the amount of about 10,800 wppm.
- At 840 °C, beta titanium begins to transform to alpha titanium on cooling through the beta-alpha transus. This transformation will be complete around 600 to 500 °C, depending on the rate of cooling.

During the transformation, tantalum is rejected from the decomposing high solubility beta phase into the remaining untransformed beta phase. This results in an elevation of the melting temperature and lowering of the beta-alpha transus temperature of the remaining beta phase (see lines marked "solidus," "liquidus," and "beta transus" in Fig. 11). Note how these temperatures are effected by the increasing tantalum content.

Since some regions, known as "retained beta phase," will have more than the average tantalum composition of 15% (because of the aforementioned segregation process), other regions of the alloy must have less than the average tantalum content. The regions with lower than average tantalum content will be those parts of the alloy that transformed to alpha titanium on cooling below the beta-alpha transus. These tantalum-depleted regions will have a depressed melting temperature and an increased alpha-beta transus temperature. The melting point of very low tantalum content titanium can be as low as 1672°C.

Such low melting range alpha-titanium regions, which are located throughout the bulk of the material and along grain boundaries, will melt during operation and provide local regions of increased electrical resistance, leading to further local overheating and mechanical separation. Recall that liquid films do not transfer shear stresses.

In summary, two complementary mechanisms are offered as explanations for the progressive cracking and microstructural changes that have been observed in titanium-15% tantalum getter wires that have failed during operation between room temperature and 1750 °C in a partial pressure of hydrogen, oxygen, nitrogen, carbon monoxide, and carbon dioxide. Both mechanisms explain how voids or cracks form at transformed alpha-titanium grain boundaries and within the grains, and how these internal discontinuities act as local "hot spots" when electrical current is passed through the getter wires.

RECOMMENDATIONS

Keeping the wires above the alpha-beta transition temperature (840 °C) during idling will prevent the precipitation of alpha titanium in the material, resulting in maintaining a more homogeneous distribution of tantalum. The addition of thermocouples to enable the monitoring of getter wire temperature would facilitate the maintenance of idling temperature above the alpha-beta transus temperature.

Cooling the getter wire slowly from operating temperature at low pressures of 10^{-3} torr would allow the desorption of hydrogen from the wire,² eliminating the formation of hydrogen porosity and, if the hydrogen is outgassed to levels below the solubility limit in alpha titanium (1840 wppm), the formation of brittle hydrides.

REFERENCES

1. Varian, Vacuum Division, Technical Bulletin, VAC 2276D, Section 6.
2. G. W. Wille, J. W. Davis, Hydrogen in Titanium Alloys (DOE/ET/520359-2), April 6, 1981.
3. R. I. Joffie, "The Physical metallurgy of Titanium," Progress in Metal Physics.
4. B. Chalmers (ed.), Permmagon Press, 1958.

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Fig. 1. Characteristic Curves. Pumping speed depends on area, conductance, sublimation rate, and pressure.

Kroll's Reagent

Mag: 100 X

Fig. 2. Unexposed material exhibiting heavily worked grains.

Kroll's Reagent

Mag: 100 X

Fig. 3. Unexposed material exhibiting recovered structure.

Kroll's Reagent

Mag: 100 X

Fig. 4. Material that operated successfully. Note the fine acicular alpha formed by a fast-cooling rate.

Kroll's Reagent

Mag: 100 X

Fig. 5. Material that operated successfully. Note the coarse acicular alpha formed by a slow-cooling rate.

Kroll's Reagent

Mag: 50 X

Fig. 6a.

Kroll's Reagent

Mag: 50 X

Fig. 6b.

Fig. 6a & b. Coarse acicular alpha. Dark spheres are voids. These voids are probably caused by the decomposition of titanium hydrides and/or hydrogen coming out of solution upon cooling from the operating temperature range of 1600 to 1750 °C down to 400 °C.

Kroll's Reagent

Mag: 50 X

Fig. 7. Photomicrograph showing precipitated alpha in grains and along grain boundaries.

Kroll's Reagent

Mag: 100 X

Fig. 8. Dendritic structure and shrinkage cavities (a) showing melting of the alpha phase (b).

Krolls Reagent

Mag: 50 X

Fig. 9. Melting of precipitated alpha at grain boundaries.

Krolls Reagent

Mag: 40 X

Fig. 10. General melting of precipitated alpha as evidenced by dendritic structure (a) and shrinkage cavities (b).

Fig. 11. Ti-Ta phase diagram.