Characterization of Mounted Hub Section
(Material Characterization)

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Evaluation Report
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PURPOSE

Identify and characterize the "hardened layer" observed at the submitted sample's fracture origin.

BACKGROUND

WL/MLSA was contacted by George W. Anderson regarding an ongoing National Transportation Safety Board (NTSB) investigation. As a result of a telephone conversation with Mr. Anderson, 13 December 1996 (Attachment 1), NTSB Metallurgist's Factual Report 96-131 was forwarded to WL/MLSA for review and comments on 24 December 1996. The report was reviewed by Larry P. Perkins and several conclusions/comments were forwarded by letter to the NTSB on 25 March 1997 (Attachment 2).

This letter identified the discussed "hardened layer" as an alpha case and suggested the following evaluations be completed to ensure an adequate identification of the cause of formation:

- Complete chemical analysis to confirm the alloy composition (believed to be Ti-6Al-4V).
- Examine other (adjacent) holes on the failed disk to determine if this condition is a one-time occurrence.
- Examine overhaul records.
- Identify alpha layer and stabilizer(s) by Auger and/or secondary ion mass spectrometry (SIMS) testing.

The NTSB submitted a mounted sample (Figure 1) to WL/MLSA for SIMS testing of the "hardened layer". This sample contained the failure origin as located in a tierod hole.

A meeting was held on 22 May 1997 to review and discuss the testing conducted to date. As a result of this meeting, a request for testing was developed (Attachment 3) that included the following:

- Complete chemical analysis.
- Metallographic examination of other (adjacent) holes on the failed disk.
- SIMS mapping and characterization of the alpha layer.

As a result of the testing requested on 22 May 1997, it was determined that cutting of the sample would be required (for further SIMS testing and bulk wet chemical analysis). WL/MLSA conducted a scanning electron microscope (SEM) examination to document the sample before cutting on 23 and 26-27 May.

On 26 May 1997, Michael L. Marx of the NTSB requested that testing be halted, the sample be returned, and a report on tests conducted to date be provided (Attachment 4). The specimen was returned without being cut.

FACTUAL DATA

The submitted sample was removed from its mount for extreme low vacuum (10^{-9} torr) SIMS analysis. Preliminary SIMS analysis was conducted between 12 and 22 May 1997. Two regions were examined on the polished surface at the edge of the tierod hole (the area containing the identified "hardened layer"). Each of these regions was mapped for oxygen and carbon. The second region was also mapped for iron and silicon. The maps generated and presented on 22 May are shown in Figures 2 and 3. The first region was found to contain elevated concentrations of oxygen and carbon to depths of 34 and 21 um from the tierod hole edge, respectively. A subsurface carbon deposit (Figure 2) was identified in this region. The second region was found to contain elevated concentrations of oxygen, carbon, and iron to depths of 139, 145, and 107 um, respectively. A subsurface carbon deposit (Figure 3) was also detected in this region. The silicon map was enhanced. Evidence of a slightly increased silicon concentration was observed in the "hardened layer", however, the enhancement introduced extraneous information into the map.

SEM and Energy Dispersive Spectroscopy (EDS) examination of the specimen was conducted on 23 and 26-27 May. Backscatter electron imaging (BEI) of the polished surface identified evidence of at least 29 cracks emanating from the tierod hole in the "hardened layer" (Figure 4). The sample was observed to be etched and contained a bulk microstructure of equiaxed alpha (dark in BEI mode) in a matrix of transformed beta containing fine acicular alpha (close-up regions 1 and 2, Figure 4). A distorted microstructural layer was observed to transition from the bulk material to the "hardened layer". The directionality of this distortion indicates a clockwise (as viewed down on the polished surface) plastic deformation of the tierod hole's surface. A small patch of higher intensity electron return was detected within the "hardened layer" (labeled A in Region 1, Figure 4).

Regions 1, 3, and 4 (Figures 5 through 7) were then EDS mapped at 10kV for K-band oxygen, silicon, aluminum, carbon, vanadium, iron, chromium, nickel, and L-band tungsten. The small patch observed to reflect higher intensity electrons (labeled A in Region 1) was found to contain an elevated concentration of iron (Figure 5). This patch was measured to be 43 um below the surface of the tierod hole. Examination of Region 3 identified elevated concentrations of oxygen, aluminum, iron, and chromium in the first 4 um, 18 um, 5 um, and 5 um of "hardened layer", respectively (Figure 6). Examination of the bulk microstructure from Region 4 identified elevated concentrations of aluminum in patches corresponding to the equiaxed alpha microstructure (Figure 7).

BEI of the tierod hole surface was conducted (Figure 8). Patches of high intensity electron return were observed in Regions 5 and 8. Smaller patches of low intensity electron return were identified in Region 5. Localized directional marring (Region 6) was observed approximately 1.8 mm from the polished surface as indicated. Several patches of low intensity (black regions) were observed on the hole surface (Region 7). A difference in surface morphology was also observed near the polished surface containing the cracks.

Regions 5 through 8 were then EDS mapped at 10kV for K-band oxygen, silicon, aluminum, carbon, vanadium, iron, chromium, nickel, and L-band tungsten. Region 5 was observed to contain patches that were found to be high in iron and chromium that corresponded to the patches of high intensity electron return (red arrows, Figure 9). The dark patches in this region were observed to contain oxygen and silicon. Examination of Region 6 identified elevated concentrations of oxygen and silicon in the patches of localized directional marring (Figure 10). Region 7 was observed to contain dark patches that were also identified as oxygen and silicon (Figure 11). The high intensity patch in Region 8 was found to contain an elevated concentration of iron (Figure 12).

A section of mounting compound that was removed from the sample, was gold coated, and was examined using EDS. The mounting compound was observed to contain carbon, oxygen, iron, aluminum, silicon, and calcium (Figure 13).

DISCUSSION(S)

Evaluation of the specimen identified that a surface etch had been conducted on the polished surface, allowing observation of the bulk microstructure and the "hardened layer" (Figure 4). Examination of the tierod hole surface also showed signs of etching, particularly near the polished surface containing the cracks (Figure 8). This surface was observed to contain a different morphology than the rest of the hole surface and is believed to have been blue acid etched.

At least 29 cracks were observed propagating from the hole/polished surface edge. The nature of these cracks could not be determined without sectioning the specimen.

EDS analysis of the bulk microstructure identified elevated concentrations of aluminum corresponding to the equiaxed alpha grains. This observation is consistent with the fact that aluminum is a substitutional alpha stablilizer. Aluminum is more soluble in the alpha phase and stabilizes alpha formation at higher temperatures. This segregation of aluminum (due to its solubility) to alpha phase was observed in this map (Figure 7).

EDS analysis of the "hardened layer" identified elevated concentrations of oxygen, aluminum, iron, and chromium. Oxygen is a known interstitial alpha stabilizer and was detected at elevated concentrations to a depth of 4 um. Titanium surfaces readily dissolve oxygen at high temperatures when exposed to air or oxidizing atmospheres. Aluminum, as mentioned, is also an alpha stabilizer and was detected at elevated concentrations to **a** depth of 18 um. The presence of elevated concentrations of oxygen and aluminum at the surface confirms the presence of an alpha case at this edge. Due to the limitations of EDS, the detectability of oxygen is far less than the detectability of aluminum. The alpha case depth is thereby estimated to at least the depth of the elevated aluminum concentration (18 um). The actual alpha case may be deeper. The limits of EDS (minimum detectability: 0.1 wt%) preclude detection of smaller concentration gradients. Iron and chromium are beta eutectiod stabilizers that promote intermetallic formation upon cooling. The presence of these two elements indicates either the possibility of intermetallic formation and/or gross material transfer/mixture with the sample during plastic deformation.

SIMS analysis also identified oxygen and iron in elevated concentrations in the alpha layer as well as an elevated concentration of carbon. Carbon, like oxygen, is an interstitial alpha stabilizer. Unlike oxygen, however, carbon does not diffuse readily and is usually found in alpha case as a result ***of*** carbon contamination during forming or machining at elevated temperature. The tierod bolt hole surface was not drilled before forming and the source of contamination must, therefore, be assumed to be drilling/boring operations. The elevated oxygen again verifies that an alpha case is present. However, estimation ***of*** its thickness (34 and 139 um measured), based upon SIMS results, must take into account that **SIMS** is sensitive to as little as 1 part per 10 million. The elevated oxygen concentration thicknesses measured may contain a nonalpha microstructure with slightly elevated oxygen concentrations as well as the alpha case.

The observed deformed microstructure under the alpha case indicates plastic deformation in a clockwise direction with respect to the polished surface. The presence of an alpha case indicates that overheating of the tierod hole occurred. The directionality of the deformation provides further evidence that a manufacturing tool (drill) played a role in formation.

Silicon and oxygen were detected at low intensity electron backscatter regions on the tierod bolt hole surface. These regions were all observed to be at "low spots" topographically. EDS analysis of the mounting compound identified the presence of both silicon and oxygen (most likely silica). It is most likely the silicon and oxygen observed in the EDS maps is trapped mounting compound.

EDS mapping of the polished surface detected a patch of iron under/within the alpha layer. Assuming this iron was not deposited on the surface during NTSB metallographic processing, it is believed this iron was transferred from a drilling **or** boring tool during an overheating condition. This resulted in alpha case formation and subsurface deformation of the tierod bolt hole. No elevated concentration of chromium was detected indicating the iron was not from a stainless or high speed steel. However, the EDS minimum detectable 0.1 weight percentage, combined with the geometry of the patch, may mask any elevated concentration of chromium. SIMS analysis of this region may better reveal the alloy type of this material. A match against drilling/boring bit material would narrow down the potential source of the alpha layer.

EDS mapping of the tierod bolt hole detected patches containing both iron and chromium. These patches on the surface have three probable sources: (1) transfer from **a** drilling or boring tool, (2) transfer from the tierod, or (3) transfer from another object after hub separation. High speed steels contain up to 13 percent chromium. SIMS testing of the potential source tools against the material identified may provide a match.

Examination of the two adjacent tierod bolt holes may provide further information as to the source of alpha formation. The detection of alpha, directional plastic deformation, or transferred iron may show a tool degradation (previous hole) progressing to a worst case tool condition (in failed hole) and improvement upon tool replacement after breakage (next hole).

CONCLUSION(S)

An oxygen-rich alpha case is present on the tierod bolthole edge. This case is approximated to be between 18 and 139 um deep.

Elevated concentrations of iron and carbon were observed in the alpha case indicating gross material transfer/contamination of the titanium surface. Contamination potentials include:

- Machine (drilling/boring) tools
- Tierod bolts
- Foreign material

An iron-rich patch of material was found in the hardened alpha layer.

Iron and chromium rich deposits were identified on the tierod hole surface.

Silicon and oxygen were also detected on the tierod hole surface. Most is likely from residual mounting compound.

RECOMMENDATION(S)

Examine the adjacent tierod holes for alpha case, plastic deformation, and iron transfer.

Conduct SIMS analysis of the subsurface iron patch to better type the alloy.

Identify tierod/drilling tool/boring tool compositions to determine the source of iron transfer and, therefore, identify the cause of overheating and alpha formation.

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