ANALYSIS OF MERCURY DIFFUSION PUMPS

by

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A paper proposed for presentation at
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ABSTRACT

Several mercury diffusion pump stages in the Tritium Purification process at the Savannah River Site (SRS) have been removed from service for scheduled preventive maintenance. These stages have been examined to determine if failure has occurred. Evidence of fatigue around the flange portion of the pump has been seen. In addition, erosion and cavitation inside the throat of the venturi tube and corrosion on the other surface of the venturi tube has been observed. Several measures are being examined in an attempt to improve the performance of these pumps. These measures, as well as the noted observations, are described.

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SUMMARY

Six stages [two machined (MP) and four electron beam (EB) welded] from the mercury diffusion pumps operating in the Tritium Purification process at SRS have been analyzed to determine their condition after nine months of usage. Several cracks were found around the necked region of the two MP stages. The EB welded stages, however, seemed to perform better in service—two of four stages showed cracking. The cracking is caused by fatigue that has been enhanced by high stresses and tritium in the flange area.

The EB welded stage appears to be a step in the right direction. Since the EB weld is a shrink fit, the surface is in compression, thereby eliminating crack propagation. In addition, shot peening has been employed to produce a compressive material surface since fatigue usually originates at the surface.

Pitting was observed down the throat of the venturi. This pitting was caused by cavitation and erosion along the length of the venturi tube.

Corrosion and pitting was seen on the exterior walls of the diffuser tubes. Stress-corrosion cracks were observed emanating from these corrosion pits. The corrosion likely occurred from the chloride ions present in the process cooling water. Shot peening is now being used in an attempt to place the outside of the diffuser tube in compression to eliminate the stress-corrosion cracking.

INTRODUCTION

The mercury diffusion pump in tritium is a three-stage, booster-ejector pump originally manufactured by Consolidated Vacuum Corporation (CVC). This pump pulls gas from the furnaces through a uranium decomposer bed to the Sprengel forepumps for further purification. The unit can increase gas pressure from microns up to approximately 30 kPa, with a maximum capacity of approximately 8000 cm³/minute (similar to most Hg vacuum pumps). Mercury is vaporized in the boiler and pushed to the top of the pump where it condenses and entrains incoming gas and flows downward through a diffuser tube or venturi. The diffuser tubes are cooled by the surrounding cooling water jackets that initially condense the mercury as it flows down the venturi [1]. The mercury vapor temperature inside the diffuser tube is around 573K and the outside temperature is about 293K, which creates a tremendous thermal gradient between the walls. Figure 1 represents one stage of the mercury diffusion booster-ejector pump.

There are two mercury diffusion pumps (237 and 232) in the Tritium Purification process at SRS. The 237 pump requires a more rigorous operation than the 232 pump because it sees tritium gas prior to purification. Once the gas reaches the 232 pump, it has already been through the uranium decomposer bed and the palladium/silver diffuser for purification. Originally, the position of the stages and their performance were thought to be independent of one another. However, because of insufficient evidence for that hypothesis, stage performance in reference to position is now being monitored.

The original mercury diffusion pump stage design incorporated a tungsten inert gas (TIG) weld to interface the flange with the diffuser tube. Failures with these stages, at the TIG weld, occurred in 1986 and 1987 after only three months of service. An investigation was initiated to determine how the life of the diffuser stages could be extended.

Finite element analyses were employed to examine the stress states that were present due to thermal stress, residual weld stress, shrink-fit weld stress, and the combination of thermal and residual weld or shrink-fit weld stresses on the stage of the mercury diffusion pump diffusers [2]. The analyses showed that the presence of principal stresses, as a result of high bending stresses, were located in the venturi tube area and the necked region for the thermal case. However, the stress analysis for the residual welding stress case predicted tensile stresses in the horizontal and necked regions of the mercury diffusion pump stage. According to the study, neither of the stress-related problems would, separately, cause any cracking through the wall. However, combining the two stresses would result in high-tensile stresses located in the necked region. From this finite element analysis, several modifications to the previous design were made. The two most recent designs included a stage machined out of a single block of steel, while the other design incorporated a shrink-fit EB weld between the top flange and the diffuser tube.

In an attempt to maintain failure-free pumps, the current preventive maintenance schedule requires the removal of the third stages after nine months of service and the first and second stages after 18 months of service. Although no service failures have occurred while using this preventive maintenance regimen, cracking has been observed. Table 1 shows the third stages examined and their properties.

PROCEDURE

All stages removed from service were tested with a dye penetrant to determine if any cracking was visible in the necked region of the stages. Once tested, cracked stages were stored in an air hood until the material had sufficiently outgassed. This was done so that sectioning could be performed without tritium contamination. Most of the flange was cut away to expose the diffuser tube region. The diffuser tubes were sectioned along their length adjacent to cracks. Once the pieces were of the workable size, stereomicroscopy and scanning electron microscopy (SEM) were used to examine the cracks in the necked region of the venturi. In addition, metallography was used to examine both crack morphology and crack depth.

Energy dispersive spectroscopy (EDS) was used to examine the extraneous debris on the inner surface of the diffuser tube as well as the corrosion product on the water side. SEM was employed to examine pitting of both the inner and outer surfaces of the stage prior to metallographic examination of the pits and cracking.

RESULTS AND DISCUSSION

Dye-penetrant checks of the first and second stages showed no cracking in the flange. Since these stages see a smaller velocity of gas and mercury flowing through them, their cyclic and tensile stresses are lower, therefore decreasing the probability of fatigue failure.

One of the more recent designs has involved machining the stages out of a single block of 304L stainless steel in an attempt to eliminate flaws that may be introduced by welding processes. Two machined third stages were removed from service, one in 1988 and one in 1990. Dye-penetrant testing revealed approximately 12 cracks around the necked portion of each stage (Figures 2 and 3). A full destructive examination of both the 1988 MP and the 1990 MP stages has been conducted.

Another design that has been examined recently is the shrink-fit, EB weld technique to connect the diffuser tube to the top flange. The shrink fit was used to place the origin of cracking (inside surface of necked region) in compression. In two of the four EB welded third stages removed from service since 1988, dye-penetrant testing revealed cracking (Figures 4 and 5).

Previous investigations by Ehrhart and Eberhard [1, 3] have attributed the mercury diffusion pump failures to liquid metal embrittlement and stress-induced cracking. They also claim that mercury globules were present in the cracks of the metallographic specimen. It is not unusual, however, that mercury droplets were found in the cracks since mercury flows through the tubing. Furthermore, any type of debris present on the parts will tend to congregate at a crack site. Upon examination of liquid metal embrittled samples, one should see a complete coating of the liquid metal. In fact, the liquid metal is in such close contact with the solid that it is generally difficult to remove. Therefore, the presence of mercury droplets in the crack is not sufficient justification for concluding liquid metal embrittlement. The present studies found no coating of mercury, but a few mercury droplets were observed. Liquid metal embrittlement has, for the most part, been ruled out as the failure mechanism.

Following the sectioning of the stages, it was observed that the cracks in all samples were approximately 2-4 cm along the length of the diffuser tube. The cracks were examined by SEM (Figure 6). It was unclear from these micrographs whether the cracking occurred intergranularly or transgranularly. In addition, the SEM of opened cracks did not reveal a crack origin or reason for fracture. Several particles were observed on the interior surface of the stages near to and around the cracks (Figure 7). The particles were rich in silicon (Si), aluminum (Al), and potassium (K), as shown by the EDS spectrum in Figure 8. Although great in number, the particles were probably not a factor in the failure of the mercury diffusion pumps. There are two possible explanations for the presence of the particles. First, Al and Si were observed during an elemental examination of a

spot-check penetrant used in the dye-penetrant testing (Figure 9). The second explanation deals with the presence of zeolite beds upstream in the process, which may provide a source of Al, Si, and K.

Cross-sections of the top portion of four mercury diffusion pump stages were cut to conduct metallographic examinations. Optical microscopy on the mercury diffusion pump stage pieces was employed to observe the cracking in the necked regions (Figures 10-12). As can be clearly seen, the cracks are mostly transgranular in nature. Propagation of the crack has occurred from the inside of the necked region toward the outside wall and is not indicative of stress-corrosion cracking. In addition, no pitting is visible at the origin of the cracking. The cracks appear to be mainly related to fatigue. They are clean, there is not much branching, and they are fairly straight. The cracks extended, at the most, one-third of the way into the sections and no through-wall cracks were observed. This is not unusual since the cyclic stress needed to cause fatigue cracking is less than the yield stress of the material. Therefore, a through-wall crack may not occur at all.

There were unusual regions observed in the metallographic section of the 1989 EB welded stage shown in Figure 12. They are small, approximately 5 μm in diameter, and appear to be ferrite. Figure 13, a longitudinal section from this same area, shows the presence of stringers (probably ferrite). Transmission electron microscopy (TEM) was employed to identify these artifacts. Particles found in the matrix were either chromium (Cr) and manganese (Mn) rich or manganese sulfide (MnS) inclusions (Figures 14 and 15). Because of problems with thinning, the particles in Figure 14 could not be identified with diffraction patterns. Since Cr is a ferrite stabilizer, it is likely that the particles are ferrite. However, because of the lack of diffraction information and the presence of Mn, ferrite cannot be confirmed.

A large degree of pitting was observed down the length of the venturi tube, primarily in the 1988 stages that were examined. The 1988 EB welded piece was pitted far more than the remaining stages. Macrographs representative of the pits that formed on the interior wall of the 1988 EB welded stage are shown in Figure 16, while SEM micrographs of the pitting are shown in Figure 17. The appearance of the pits is typical of cavitation, which involves a material buildup along a surface. The pressure is increased at the surface into a range such that a cavity is formed from the force of the eventual pressure release. As indicated by the micrographs, the pits are approximately 62.5 μm deep and numerous.

SEM micrographs representative of the pitting in the 1988 MP piece can be seen in Figure 18. The pits in the MP piece typify erosion pitting, where erosion is defined as the removal of surface material due to numerous impacts of the surface from solid or liquid particles.

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SEM micrographs representative of the pitting in the 1988 MP piece can be seen in Figure 18. The pits in the MP piece typify erosion pitting, where erosion is defined as the removal of surface material due to numerous impacts of the surface from solid or liquid particles.

The pits in both the 1988 EB piece and the 1988 MP piece had several particles in them that were rich in Al, Si, and K. However, the particles are not attributed to the cracking in the pump or the pitting. They are believed to be from the process system.

It is evident from the metallography performed on the cross section of the 1988 EB part that, although the pits were very prominent and visible, none of the interior wall pits propagated cracks (Figure 19). This fact is encouraging since the interior of the mercury diffusion pump stage should be in compression from the shrink-fit weld. The pits on the 1988 MP stage (Figure 20) are not as noticeable by metallography as those from the 1988 EB welded stage.

The fact that no cracks were found propagating from the cavitation/erosion pits in 1988 MP and 1988 EB and that pitting was not evident of the interior walls of the two 1989 EB welded stages provides sound reasoning to assume that the pitting will not contribute to the failure of the pump.

There were, however, several corrosion pits observed on the exterior wall of most diffuser tubes examined. The pits acted as crack initiators in the 1988 EB and 1988 MP stages (Figures 19-22). The cracking that was observed typifies stress-corrosion cracking. Chloride ions present in the cooling water are suspected to be the corrosive medium. To combat this problem, the outer walls are now shot peened to place the surface in compression. Stage 1990 EB was shot peened on the outside of the diffuser tube and it did not exhibit any corrosion.

CONCLUSIONS

Table 2 shows the results from the stage analyses. From the course of these analyses, several conclusions can be made. The EB shrink-fit weld procedure is necessary since it appears to have eliminated much of the tensile stress associated with the mercury diffusion pumps. A decreased number of cracks present around the necked portion of the diffuser tube indicates that the design and strength of the material is a step in the right direction. However, it is important to note that material quality is a necessity to having failure-free pumps. Machining the stages out of a single piece of metal will be eliminated because of the presence of numerous cracks observed in the examined stages.

Shot peening the entire surface of the flange also seems to have an advantageous effect toward eliminating the cracks in the mercury diffusion pump stages. However, the shot peening must be conducted on the entire flange rather than from the EB weld outward, or not at all. Stage 1990 EB, the only stage that was shot peened on the entire flange, did not have any cracks in the necked region of the diffuser tube. However, of the three stages that were not shot peened on the entire flange, 1988 EB, 1989 EB, and 1989 EB (cracked), two had cracks in the necked region of the diffuser.

Shot peening produces compressive residual stresses on the surface by the introduction of a high-velocity stream of shot to the surface. With the material surface in compression, the cracks are inhibited. However, the cracks will propagate once the stress is overcome. Future analyses of third stages will continue to provide information on the success of the shot peening.

No cracks were observed propagating from the erosion/cavitation pits in the 1988 MP stage and the 1988 EB stage. In addition, no significant cavitation or erosion was observed along the inside length of the diffuser tubes from the remaining four stages examined. Therefore, neither the cavitation nor the erosion should be deleterious to the life of the mercury diffusion pump stages.

Finally, shot peening the outside of the diffuser tube has been used to eliminate the stress-corrosion pitting and crack propagation associated with the chloride ions. Since shot peening has been shown to provide protection against corrosion and crack propagation, it will continue to be used on future mercury diffusion pump stages.

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ACKNOWLEDGMENT

The information contained in this paper was developed during the course of work done under Contract No. DE-AC09-89 SR18035 with the U.S. Department of Energy.

Table 1. Mercury Diffusion Pump Stage Information

<u>Name</u>	<u>Stage</u>	<u>Start Date Examine</u>	<u>How Fabricated</u>	<u>Pump Position</u>
1988 EB	3rd	10/88	EB welded	—
1988 MP	3rd	10/88	machined from single block	—
1989 EB (cracked)	3rd	10/89	EB welded and shot peened from weld to outer flange	—
1989 EB	3rd	10/89	EB welded and shot peened from weld to outer flange	—
1990 EB	3rd	7/90	EB welded and shot peened on all flange and outer venturi	237
1990 MP	3rd	7/90	machined from single block	232

Table 2. Results from Mercury Diffusion Pump Stage Analyses

<u>Name</u>	<u>Cracks in Flange</u>	<u>Pits Inside Diffuser</u>	<u>Pits Outside Diffuser</u>	<u>Shot Peen</u>
1988 EB	1	yes—cavitation	yes—corrosion and crack propagation	no
1988 MP	— 12	yes—erosion	yes—corrosion and crack propagation	no
1989 EB (cracked)	— 6	no	corrosion product/no cracking	yes—EB weld out on flange
1989 EB	no	no	corrosion product/no cracking	yes—EB weld out on flange
1990 EB	no	no	no pits or corrosion product	yes—all flange surface and outside diffuser
1990 MP	— 12	no	some corrosion/no cracking	no

M91aug001.01

Figure 1. Schematic of CVC Third Stage

Figure 2. Dye-Penetrant Test from 1988 MP

Figure 3. Dye-Penetrant Test from 1990 MP

Figure 4. Dye-Penetrant Test from 1988 EE

Figure 5. Dye-Penetrant Test from 1989 EB

Figure 6. SEM Micrograph of Opened Crack in 1989 EB Welded Stage

Figure 7. SEM Micrograph Representative of Particles on Mercury Diffusion Pump Stage

SERIES II
Cursor: 0.038keV • 0

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Figure 8. EDS of Particles on Mercury Diffusion Pump Stage

SERIES II
Cursor: 0.000keV = 0

ED 29-JUN-88 15:34

Figure 9. EDS of Dye-Penetrant Check

Figure 10. (a) Flange Region of 1988 MP
(b) Higher Magnification of Crack in (a)

Figure 11. (a) Flange Region of 1988 EB Welded Stage
(b) Higher Magnification of Crack in (a)

Figure 12. Transverse Section of Crack in 1989 EB Sample
(Note the outlined artifacts in the material.)

Figure 13. Longitudinal Section of Crack in 1899 EB Stage

Figure 14. Cr and Mn Rich Particle in Matrix

Figure 15. MnS Inclusions in Matrix

Figure 16. Pits Inside Diffuser Tube from 1988 EB

Figure 17. SEM Micrographs of Pits from Inside 1988 EB

Figure 18. SEM Micrographs of Pits Inside 1988 MP

Figure 19. Metallographic Section of Diffuser Tube from 1988 EB

Figure 20. Metallographic Section of Diffuser Tube from 1988 MP

Figure 21. Stress Corrosion Cracks Propagating from Exterior Pits from 1988 EB

Figure 22. Stress Corrosion Cracks Propagating from Exterior Pits from 1988 MP

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