FAILURE ANALYSIS AS AN ESSENTIAL COMPONENT OF AN SOFC DEVELOPMENT PROGRAM

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ABSTRACT

Post-test analysis provides the stack and materials development teams with valuable feedback after each test. Prototype systems, stacks and single cells are taken apart following a standard procedure to capture all relevant information. Those observations lead to a root cause analysis of the failure of either stack or single cell. Hence a loop is created, linking every activity of the program from research and development, pilot plant production, testing and back to research and development. Failure analysis tools Failure Modes and Effect Analysis (FMEA) and Fault Tree Analysis (FTA) provide support for root cause analysis. The analysis itself is performed by a standard set of autopsy procedures, scanning electron microscopy (SEM) and possibly x-ray diffraction (XRD). A collection of information is then grouped and trends are noted and related to the performance of cell, stack or prototype systems. Learning and recommendations are recorded and implemented. Failure analysis is the activity that closes the loop of the development program cycle.

INTRODUCTION

Versa Power Systems’ SOFC development team has been working in a close loop as an integrated group to alleviate any communication gaps between functional departments, to share the learning, to implement innovative ideas quickly and to ease problem solving (Figure 1). As a component of this program, post-test analysis activity is essential in communicating root cause analysis of failures to the testing team, stack developers and pilot-plant production team (1).

The SOFC stack is complex. It contains components with different shapes and designs and of different materials, ceramics and metals. It operates at a high temperature in dual environment, air and reducing fuel (H₂ or natural gas). Stack operation generates power and an exhaust including steam (2). The stack contributors are summarized in the list below:

- Mechanical design, fabrication process of components and stack assembly.
- Material selection for good integrity and mechanical match.
- Testing conditions.
Failures recorded in such complex development program can be catastrophic occurring at the infancy stage or end of life type occurring after a "long" degradation. Hundreds of stacks were autopsied in the last 4 years. The failure analysis activity has changed during those years and had to adapt to new types of failures. Two stages with different types of failure can be distinguished. The first type of failures mainly occurred in the early years of the development program. Corrective actions from those failures were needed to optimize processes and to provide appropriate training. The second type of failure is part of engineering learning stage and research and development activity. Examples from each type of failure types are presented here.

DESCRIPTION OF THE ACTIVITY

The analysis itself is performed by a standard set of autopsy procedures, scanning electron microscopy (SEM) and possibly x-ray diffraction (XRD). A collection of information is then grouped and trends are noted and related to the performance of cell, stack or prototype systems. A remedy system was also implemented to follow corrective action implementation and to record all catastrophic failures and their respective remedy.

Failure Analysis Tools

Failure analysis tools; Cause-Effect diagram, Failure Modes and Effect Analysis (FMEA) and Fault Tree Analysis (FTA) have been developed to provide support for root cause analysis (3). The Cause Effect or fishbone diagram is an overall listing of possible causes and their effects leading to a low performance stack. The FMEA is split by components and their respective functions. The FMEA is populated with all possible causes not necessarily observed in actual stacks. The FTAs have been written for specific failures. They have been useful in directing analysis towards possible causes and finally determine the root cause(s) of that particular failure. This methodology was adopted to solve system unit failures and failure trends in stacks.

To illustrate the use of the above tools, an example is presented. A system unit failed, generating only low power. The data history read a malfunction of the reformer. The
observation of the catalyst bed revealed sintering and plugging of the catalyst beads. There was carbon formation in the reformer. An FTA was written to find out the root cause of carbon formation in the reformer and failing the system. For each possible cause, a method or technique of analysis was suggested. Figure 2 presents only a partial view of the Fault Tree written for this example. After quick analysis, it was found out that the flow control of water was out of order. The part was quickly replaced and the reformer cleaned and filled with new catalyst to enable system restart. Also a new procedure was created for calibration and instrumentation check before system assembly. That type of failure never happened again.

![Carbon Formation In Reformer](image)

**Figure 2. Partial view of Fault Tree Analysis for carbon formation in reformer.**

**Standard Analysis**

After each test, a post-test analysis is performed to learn about the material condition, the component degradation and to feedback the observation and their relation with performance to test owner and all stakeholders. Each individual component is photographed and recorded (Figure 3). This method provides a quick feedback for a correction to a faulty process, a missing item in a procedure, a need for training for individuals, a design or component fabrication improvement or testing conditions modification. In early years of development, this method was mainly looking at catastrophic failures and was helpful in eliminating most of the root causes.

In the present mature years of development, this method provides a quick response to designers, researchers and testers for their test and helps them in their program. The information related to contact, interconnect oxidation and leakage is all recorded during this standard autopsy and related to cell and stack performance and testing conditions. Conclusion (possibly root cause of failure) and recommendations are the results of the autopsy.
Catastrophic Failures

In early stage of the development program, several incidents leading to catastrophic failures did occur due to lack of training of staff, undeveloped procedures, wrong material selection or non-optimized design. To illustrate that period of the development program two examples are presented:

- Braze overflow in interconnect flow channels: The early interconnects were made of brazed plates. The brazing process was critical in making good repeatable interconnect parts. From the stack autopsy and post-test analysis of stacks, it was found that cells with high degradation and bad performance in high utilization test had blocked flow channels (Figure 4). The material blocking the channel was brazing material overflowing. A change in the brazing process as well a more rigorous inspection of the interconnects after brazing was immediately implemented. There was no longer failure of stacks due to defective interconnect having blocked channels.

- A series of stacks failed identically, low OCV cells with low performance during utilization. In post-test analysis, those cells were usually cracked. The crack shape was round and very symmetrical (Figure 5). High leak marks were also found on layers with cracked cells. Parallel investigations were launched to check every component composition and dimension. It was found that a metallic plate was of the wrong thickness leading to mismatch between components on that layer inducing unbalancing loading on the cell till breakage. Also the unbalanced loading did not allow proper compression on seals explaining the high leakage. QC steps were immediately introduced before assembly to check every component dimension even if it feels redundant until confidence in the process is reestablished.
Successful tests or long term stack tests were thoroughly analyzed. As an illustration to this method, some findings, on the analysis of a stack run for 6700 hours including 4 full thermal cycles, are presented. The studied stack was filled with epoxy and let it cure before cutting it in a number of “towers”. Epoxy of a whole stack will keep the integrality of every component layers and their interface. Each tower includes all individual layers, contacts and interfaces (Figure 6). The “tower” is polished before being analyzed in scanning electron microscope (SEM) and by energy dispersive spectroscopy (EDS) as presented in Figure 7.

The location of the towers to analyze was chosen to have a complete map of oxidation and contact in the stack, layer to layer, from fuel inlet to fuel out and from air inlet to air.
outlet. Measurements of oxide thickness on cathode side and its composition were mapped throughout the stack Fig. 6. The shaded squares are the prepared towers for analysis (Figure 8). Trends were related with stack and individual cell performance after 6700 hours and 4 thermal cycles.

Figure 6. Polished tower sample.

Figure 7. SEM overview of stack layers.

Figure 8. Mapping of oxide on cathode side of interconnects.

Overall stack oxidation was not severe for such long test duration. Numerous locations have very isolated oxides. However there are quite a few areas with thick oxides over 150 microns. The overall trend revealed that the tower #13 which is located in the center of the layer is the worst and particularly for the middle cells. This observation matches perfectly with the performance of those cells. They were degrading more rapidly than others at the end of the test. A thermal management solution has to be proposed to take the heat out of the middle of the stack and allow longer life for those cells. Wherever there was a thick oxide, the contact interface at the cathode side of the interconnect was poor explaining the high rate of degradation as seen below in Figure 9.
Figure 9. Mapping of contact to cathode side of interconnects.

The EDS (Figure 10) also revealed that those thick oxides are rich in iron at the opposite of the thin oxides chromium rich and more conductive (4).

Figure 10. EDS analysis of oxides on cathode side of interconnect.

CONCLUSIONS

Failure analysis activity evolved from autopsy activity in early years to a post-test analysis in the mature recent years. The significance of the information collected and feedback into the research and development program was illustrated in few examples. Such information loop helped the development program in reaching the maturity stage with
better component quality into the stacks, longer life stacks, higher power density and less leakage.

The post-test analysis goes deeper in determining the modes of failure of stacks and in the understanding of the root cause of cell and stack behavior. The scientific approach in analyzing the successful stacks of the mature stage of the development program, using materials analysis techniques and failure analysis tools, helped in shrinking the development time by providing valuable data down to the microscopic level.

There are still challenges down the road before reaching the commercialization stage of the development program. Post-test analysis will keep identifying modes of failures of stacks and providing root cause analysis and recommendation to developers and designers.

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