Ames Laboratory
Lessons Learned – Ball Mill Explosion
June, 2015

Lesson ID: Ball Mill LL 2015-1

Title: Ball Mill Explosion – Failure to Identify Incompatible Materials and Follow Conduct of Operations/Research Work Planning and Control Protocols

Originating Organization: Ames Laboratory – Iowa State University

Date: August 17, 2015 **Contact Name/Phone #:** Sean Whalen/515-294-4965

Classifier: **Reviewer:**

Statement: Some metal powders react violently with halogenated solvents. In sealed vessels an extreme increase in heat and pressure may occur, resulting in vessel rupture or explosion. Mixing aluminum powder with a fluorinated solvent was a root cause of the explosion of the ball mill, similar reactions could occur with these families of materials.

Personnel must thoroughly evaluate chemical incompatibility when introducing new materials and chemicals into research procedures. Adequate hazard analysis was not performed prior to changing the procedure, and subject matter experts were not included in the evaluation of the risk associated with the decision to add a new chemical. Conduct of research authorizations (Ames Laboratory Readiness Review process) and SOPs were not sufficiently robust to require detailed hazard evaluation of new inputs.

Discussion: On June 18, 2015, a catastrophic vessel failure occurred in a chemical fume hood in Spedding Hall, laboratory room 246, resulting in significant damage to the fume hood and associated equipment. At approximately 12:05 p.m., a laboratory assistant began a ball milling process involving two in-house constructed pressure vessels containing steel milling balls, 3 grams of aluminum-manganese (Al_4Mn) powder and approximately 300 bar of hydrogen gas. One of the vials contained 5 ml of Vertrel® XF (decafluoropentane, $C_5F_{10}H_2$), and the other vial contained 5 ml of diethyl ether $(C_2H_5)_2O$. After the ball mill was loaded and started, it was programmed to operate for a run of 20 total hours, with 5-minute rest periods every 60 minutes. At approximately 12:45 p.m., a loud noise was heard from the hallway. Upon investigation broken glass was discovered in the hallway and debris was observed throughout the laboratory. A damaged chemical fume hood was discovered in the southeast corner of the laboratory. An emergency call was made to Ames Laboratory Security, who contacted Environment, Safety, Health, and Assurance (ESH&A) and Facilities and Engineering Services (F&ES) for assistance. ESH&A and F&ES responded, the area was secured with caution barrier tape, and cautious investigation and response activities were initiated.

The research group had been conducting pressurized hydrogen/metal powder ball milling for several years with no previous incidents. Ball milling utilizes motion and impact to finely divide materials, sometimes at ambient pressure, sometimes at elevated pressure. A powder sample is sealed in a metal vessel containing metallic or ceramic balls and the vessel is rotated at variable speeds and directions, causing the balls to mechanically pulverize the material. Organic liquids are routinely used to facilitate the milling process and prevent powders from clumping and cold welding. A fluorinated compound, Vertrel®XF, had only recently been added to the process. Vertrel® XF is in the same chemical family as Teflon, and is virtually nonflammable and largely inert. It was discovered during post event investigation that halogenated liquids, and especially fluorinated compounds, can be highly reactive with metal powders, specifically aluminum. During the ball milling the Vertrel®XF reacted exothermically with the aluminum to create aluminum trifluoride (AlF_3), generating a rapid increase in pressure and heat, causing the ball mill vial to rupture and igniting the hydrogen in a secondary explosion.

This was a significant near-miss event. If the laboratory had been occupied at the time of the explosion, a serious injury or fatality could have occurred.

Analysis: Work authorization for the research process High Capacity Hydrogen Storage Materials had been completed and the activity approved through the Ames Laboratory Readiness Review process. The group had conducted multiple runs of this procedure without incident, and had transitioned from a wet chemistry process using solvents to mechanical milling several years ago. The activity description still stated that organic solvents would be used, but the hazard management statements no longer referred to solvent use. Because of this lack of clarity in the documentation, the addition of Vertrel® XF was considered by the research group to be within the approved scope of the activity, and the change did not trigger a hold point. Safety Data Sheets, product literature, and peer research were reviewed by the research group prior to inclusion of the Vertrel, but the potential incompatibility with metal powders, specifically aluminum, was not identified.

The root causes for the event include: failure to identify the potential for a hazardous reaction to occur with the introduction of a chemically incompatible fluorine-bearing organic processing fluid, lack of a measured experimental plan given the unknowns, failure to consider impact of failure modes (what ifs) in the design of the equipment and facilities, and failure to provide barrier protection for personnel when required. Contributing causes include: lack of diverse and independent expertise involved in the readiness review process, unclear process for management and ownership of changes within the readiness review process, and no evidence of work observation associated with this activity.

Actions: Ball milling and the use of halogenated solvents with metal powders were suspended immediately after the event, and an extent-of-condition review was initiated for high-pressure and higher hazard activities. The Laboratory held three all-hands meetings to discuss the event and emphasize the need for safety and rigor in research and work activities. A joint DOE, Contractor, and Ames Laboratory team completed a comprehensive investigation, which identified the root and contributing causes listed above.

In response to this event and other identified opportunities to strengthen conduct of operations/research, Ames Laboratory is strengthening its current Laboratory wide strategic planning to further integrate science, operations, and safety. A major component is the development and implementation of an ESH&A Strategic Plan, of which the Safety Performance Improvement Plan (SPIP) is a key tool. Improvements focus on four main areas with short, intermediate, and long term actions: Work Planning and Control, Contractor Assurance, Engagement with Laboratory Personnel, and Expertise and Continuing Education. The Laboratory is currently completing additional Readiness Review training for all users, reevaluating hazard analysis and risk assessment for high pressure and higher hazard activities, and increasing engagement with staff.

Savings: Long-term cost saving will be realized through the prevention of injuries, accidents, and property damage.

Keywords: Explosion, incompatible chemicals, fluorinated, halogenated, aluminum, metal powder, ball mill, work planning, change, hold point.

References: SC—AMSO-AMES-AMES-2015-0001

Priority Description: Yellow/Caution

Work Functions: Conduct of Operations – General, Conduct of Operations – Work Planning, Occupational Safety and Health – General.

ISM Code(s): Define Work, Analyze Hazards, Develop/Implement Controls, Perform Work

Hazards: Fire/Smoke/NFPA, Pressurized Systems

Validator:

Attachments: Images
Ames Laboratory, Iowa State University, Room 246 Spedding Hall.

An intact ball mill machine (white) is located to the right. Destroyed ball mill (black and silver) with remaining undamaged pressure vial is located on left.