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Ferritic Steel Melt and FLiBe/Steel Experiment

Melting Ferritic Steel

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Abstract

In preparation for developing a Z-pinch IFE power plant, the interaction of ferritic steel with the coolant, FLiBe, must be explored. Sandia National Laboratories Fusion Technology Department was asked to drop molten ferritic steel and FLiBe in a vacuum system and determine the gas byproducts and ability to recycle the steel. We tried various methods of resistive heating of ferritic steel using available power supplies and easily obtained heaters. Although we could melt the steel, we could not cause a drop to fall. This report describes the various experiments that were performed and includes some suggestions and materials needed to be successful. Although the steel was easily melted, it was not possible to drip the molten steel into a FLiBe pool Levitation melting of the drop is likely to be more successful.

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Introduction

The current Z pinch IFE model includes a low-activation ferritic steel transmission line that must be recycled after it melts and falls into a molten coolant, FLiBe. FLiBe is a salt made of some highly corrosive components, and ferritic steel may or may not stand up to exposure to this molten salt. Low corrosion stainless steel is often used with materials such as FLiBe, but the alloy used in the transmission line needs to be optimized for low activation rather than low corrosion. The reaction of the molten steel and molten salt is unknown. However, one advantage of the conditions of the Z-pinch IFE chamber is that it is under vacuum, as water in the mixture is likely to release highly corrosive hydrofluoric acid gas. The objective of this project is to drop molten ferritic steel into a container of molten FLiBe to determine the reaction products in terms of evolved gases and ascertain the ability to recycle the steel.

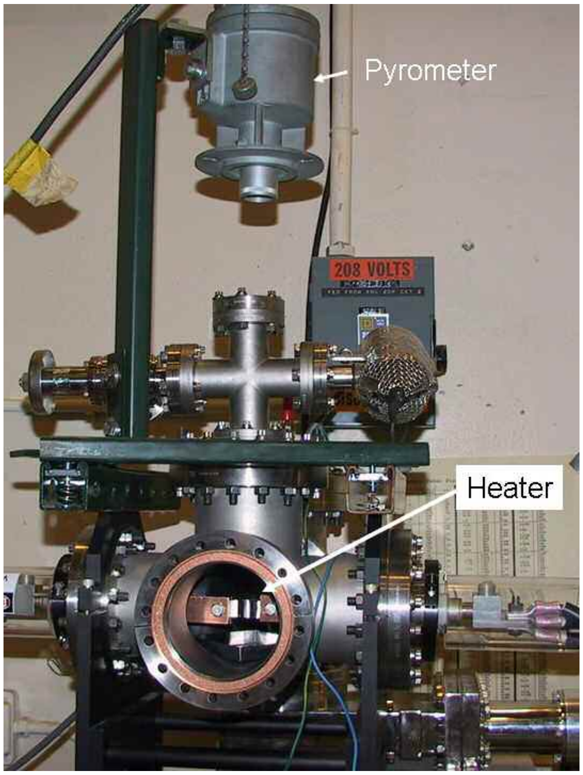
The Fusion Technology Department has studied some liquids that have potential for use as first walls in fusion reactors for magnetic fusion energy projects. These materials included lithium, gallium, tin, tin-lithium, FLiNaBe (a combination of the salts LiF, BeF_2, and NaF) and FLiKBe (a combination of LiF, KF, and BeF_2. These materials have relatively low (<500º C) melting points and were molten using vacuum compatible ceramic resistive heaters under a stainless steel crucible. The materials were melted in a vacuum system to reduce their reaction to oxygen and nitrogen. Although this heater and crucible could be used to melt FLiBe with a melting point of ~460 ºC, it cannot be used for melting ferritic steel with a melting point of ~1425º C..

The melting and dripping of molten steel was more difficult than originally anticipated. As dropping molten steel was not done by the Fusion Technology Department before, we tackled this problem first. It requires a heater that could go to higher temperature and it used a high temperature crucible. A second concern regarding this experiment was dropping the molten steel into a molten salt bath. Besides resistive heaters, the other possibilities considered were: induction heating and electron gun heating. Induction heaters have the advantage of being able to levitate material as they heat it—potentially producing an ideal situation of a small amount of molten steel that can be dropped when the levitating magnetic field is turned off. We did not pursue this given the limited time, the cost of obtaining a levitation heater and the added health and safety problems of a high frequency power supply. An electron gun can easily heat a material that is electrically grounded, but once this is done, getting the molten material to drop is difficult.

We planned to melt the steel in vacuum evaporation crucibles and drop it through a hole drilled in the bottom of aluminum oxide crucibles. Originally the steel would be larger than the hole, but as it melts, we believed that it could drip through the hole. The heaters were made of tantalum sheet or tungsten wire. The steel could not be melted directly on the tantalum or tungsten heater because in liquid form, the steel would alloy with the heaters. As iron does not react with aluminum oxide, the steel would not alloy or wet the crucibles. Aluminum oxide crucibles are also compatible with lithium fluoride, one of the major components of the FLiBe. We did not succeed with this method because the crucible/heater combination was too small to allow the drop to fall out of the crucible and larger heaters were too inefficient. Despite the difficulties and expense of an induction heater, we now believe that induction heating would produce more reliable drops of steel, although there may be some difficulties with this method also. For example, placing the steel into the levitation heater while in a vacuum will require an additional mechanical feed that needs to be non-conducting and can be taken out of the way to the molten drop.

Description of steel drop experiments and results

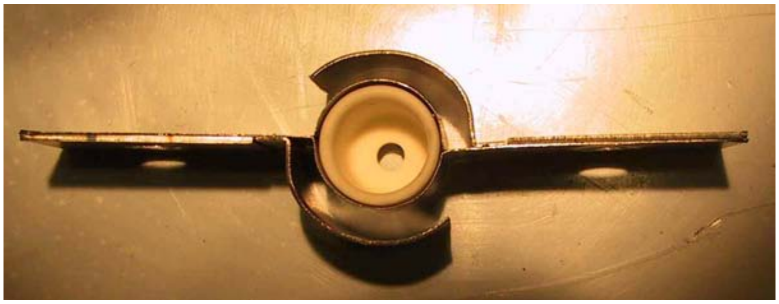
All melts were performed in a small vacuum chamber at vacuum levels below 10^{-5} Pa. We pumped the chamber with a small turbo pump and measured the vacuum with an ion gage. Temperatures of the crucible were measured initially with a type C thermocouple, but it was found that the thermocouple was picking up the current from the heater when it was trapped between the crucible and heater. Temperatures were also measured with a single color spot pyrometer to measure the metal temperature. The pyrometer viewed the ferritic steel through a sapphire window. In general, when the steel melted, the sapphire window was coated with a metallic coating. The pyrometer signal then dropped to zero. Another function of melting was the increase in vacuum pressure typically from 10^{-6} Pa to 10^{-4} Pa. A video camera recorded the side view of the crucible through a Pyrex window mounted on the side of the chamber. The vacuum chamber and pyrometer are shown in Figure 1. In this picture the power feeds are on opposite sides of the vacuum chamber. This arrangement is not optimal because the electrodes heat up and expand, compressing the heater in between them.



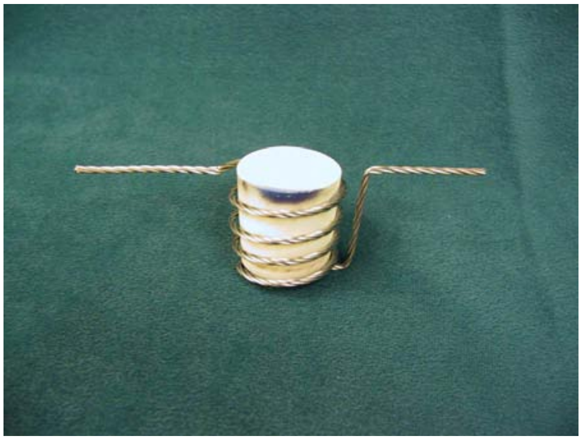
- Figure 1. Experimental set up for resistive heating.

For the experiment, Ronald Klueh at Oak Ridge National Laboratory provided a small piece of F82H ferritic steel [1]. This steel has been studied by several laboratories for strength and activation [2, 3]. It was not possible to determine the hole size needed to melt and drop the steel using gravity to overcome surface tension because information on the surface tension and viscosity was not available.

We tried two types of heaters, a tantalum heater made of a foil that is shaped into a cylinder, and a wire basket heater. Both of these heaters are designed to hold a crucible. Heaters and crucibles were ordered from R. D. Mathis Company [4], suppliers of vacuum evaporation heaters. Two sizes of crucible were considered: the small crucible with a diameter of 12.5 mm and height of 12.5 mm, which holds about 1cc and the large crucible, with a diameter of 25 mm, and height of 25 mm, which holds about 5 cc. The 5 cc crucible was planned for the FLiBe and the 1 cc crucible for the stainless steel. Two types of heaters were obtained, Ta heaters with niobium heat shields as shown in Figure 2, and basket wire heaters as shown in Figure 2. R. D. Mathis provides a source power requirement table that is partially reproduced in Table 1. The temperature is the temperature of the heater itself, and does not include the crucible or any contents of the crucible. From this table, we would need to provide 191 amps to obtain 1600^{o} C from the small Ta heater. The temperature should be a linear function of current.



- Figure 2. Small partially heat-shielded Ta heater and crucible.



- Figure 3. Tungsten wire basket heater and large crucible.

- Table 1. Power Table for RD Mathis heaters

Heater type	Volts	Current (A)	Power (W)	Temperature (ºC)
CH-11 Small Ta heater	1.7	191	325	1600
CH-13 Large Ta heater	2.19	339	742	1600
B-10 W Wire basket	9.76	71	693	1800

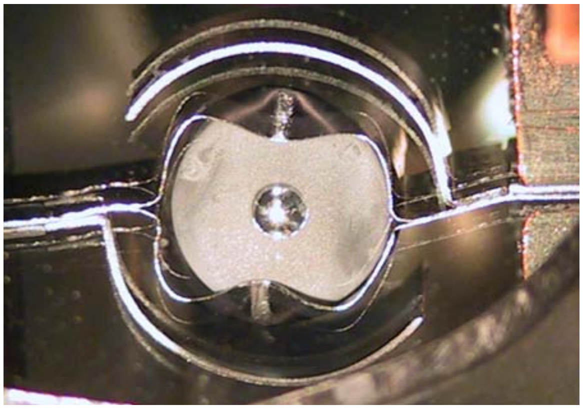
Three 100 Adc power supplies connected in parallel provided the current needed for the small heater. The small Ta heater melted the steel when the current was approximately 290 A, much higher than the current that is listed in Table 1 for 1600 ºC. This additional heat was probably required to heat the crucible and sample material. The larger Ta heater was reserved for melting the salt because it required too much current to melt steel.

The heat from the Ta heater was not confined to the crucible despite the heat shields that considerably reduced the amount of radiant heat. It was necessary was to cut off about ¼” of the heat shield wings. When the heater was hot, the heat shield tended to expand and short out to the heater. Heat conducted from the heater also increased the heat load on the electrode feed through. At first the electrodes were made of 6.45 cm^2 cross section bars equipped with slits to hold the heater, but heat was conducted to the cables outside the vacuum, causing them to overheat. To minimize heating the electrodes, currents were increased rapidly and then turned off if the chamber or cables were overheating before the steel melted. If the current was not high enough to melt the steel, the chamber cooled, and then the currents were increased rapidly to a higher level. This was repeated until the current necessary to melt the steel was determined.

The small crucible with the Ta heater was used first with a 6.25 mm diameter hole. The steel melted well in the small crucible, but would not drip through the hole. The steel did protrude through the 6.25 mm hole, but it made contact with the Ta heater, alloyed, and quickly solidified. The drop was firmly attached to the heater as shown in Figure 4. This happened because the edge of the heater was too close to the hole in the crucible.

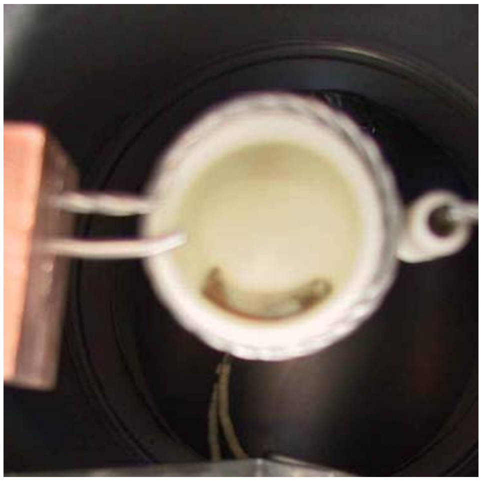
- Figure 4. Bottom of crucible and heater showing ferritic steel partway through 6.25 mm hole in crucible and touching heater

A smaller 3mm hole was tried also, but the surface tension was too high to allow the steel to flow through this hole as shown in Figure 5. Discussions ensued concerning the problem of dropping the molten steel with Michael Hosking of the Joining and Coating Department and James Maroone of Materials and Process Sciences Center, both at Sandia National Laboratories. They suggested that we clean the metal to reduce oxides and add more metal to the crucible to increase the force to push the drop out of the crucible. Their experiences on dropping molten steel have been to melt the steel and then tip the crucible or to have a large pot with a remote flange on the bottom. The other method that they had used was to inductively heat the melt and levitate it also using magnetic fields and then drop the metal by turning off the fields. Unfortunately the heater used for the induction experiment is no longer available.



- Figure 5. Bottom of crucible and heater showing ferritic steel partly through 3mm hole

Our method was modified to melt the steel without making it drop through a hole. We tried to position the basket heater and larger crucible at an angle so that a piece of steel could be placed inside the crucible and roll out when the steel melted. This also was not successful because there was no way to keep the steel in place while closing up the vacuum chamber. Small vibrations while bolting the vacuum chamber together shook the steel out of the crucible.



- Figure 6. Side view of basket heater positioned at an angle so molten steel would drop out.

Direct melting of the steel was also attempted by connecting two ends of a stainless steel welding rod to electrodes and increasing the current until the rod melted. Unfortunately, no steel dropped, the wire heated and melted, but wicked back on itself rather than dropping. This melting required about 40 amps for 2.5 min. No drops were observed on a tray placed under the wire during the melt.

To reduce the heat load in the vacuum chamber and on the vacuum feed through, the opposing feeds that are shown in Figure 1 were replaced with two water cooled feeds on a single flange. This reduces the effect of crushing the heater in two ways, 1) the electrodes elongate in the same direction and 2) the electrodes are actively cooled to minimize expansion.

We then had a hole drilled in the outer edge of the bottom of the crucible so that we could use the basket heater with the hole and avoid the wire that is placed across the bottom of the crucible. The basket was tipped slightly so that the lowest point of the crucible was located at the hole and the steel drop would avoid the bottom wire. The basket heater was unshielded, so stray radiation heated the vacuum chamber, cracking a Pyrex window. During this experiment, the steel did not melt before the window cracked. A flange containing a shutter was installed in front of a new window so it could be closed during much of the heating phase. In short, we discovered that the basket heater radiated too much heat and the heaters with the heat shields were much better at heating the crucible with a minimum of radiation loss to the vacuum chamber.

The small Ta heater was then used with the same crucible that was used earlier. This crucible had a 3 mm hole and some ferritic steel already melted in it. We added a rod of stainless steel (Figure 7) to the crucible and heated it using the water-cooled feed through. Although the rod melted, it would not drip out (Figure 8). On inspection we found that the crucible had cracked. The piece of ferritic steel that had melted in the crucible in an earlier experiment had probably expanded on heating more than the crucible and cracked the crucible. Some of the steel shorted the Ta heater. We learned that each drop must come from a separate crucible because any reheat of a crucible with a resolidified melt could cause a crack.

- Figure 7. Steel rod in small Ta heater before heating

- Figure 8. Molten steel rod in small Ta heater after melting.

Summary of molten steel work

The procedure of producing a drop of molten steel that can fall into a molten salt bath is not straight forward using resistive heating. Gravity did not overcome the surface tension and viscosity of molten steel so dripping the steel through a hole in a crucible was unsuccessful. Tipping the crucible and pouring the metal is also difficult given the necessity of high currents that require large diameter electrical feeds and perhaps water cooling. Coaxing a drop of steel through a hole in a crucible may require a heated piston of heat resistant material, and the crucible can only be used once per drop because each time steel is left in the crucible, expansion during the next heat will crack the crucible. We do not suggest more work in this direction. An alternate procedure, levitation melting, is discussed in the next section.

Levitation melting

Induction heating of metals is a method of heating material using radio frequency ac currents in a coil. The coil surrounds the material to be heated, but does not need to touch the material being heated. The currents in the coil induce eddy currents in the metal, which heat the metal. Small pieces of metal can be levitation heated, using a specially shaped coil to oppose the force of gravity. The metal can be held in place in the air or in vacuum with electromagnetic fields generated by the same coils that are heating the material.. The molten metal will drop when the coils are turned off. Since the molten metal radiates a lot of heat, the coils need to be cooled.

We have found two sources for induction heating power supplies. These suppliers also provide coils for levitation melting. We have contacted both of these companies for information on how we could accomplish the task of dropping steel into a bath and they have responded with either formal or informal quotes. Ameritherm, Inc [5] sells a small unit for approximately $10,000 that includes the power supply, coil, and cooling system for the coils. Lepel Company [6] has larger units that cost about twice as much.

Difficulties of performing a melt in a vacuum system include initially suspending the steel in the coil and shielding the coils from the heat. Both of the companies listed above have videos of levitation melting in which they use a glass or quartz rod to suspend a piece of metal in the coil. A mechanical arm will be required to do this step. We may be able to avoid needing a heat shield for the coil if we can sufficiently cool the coils with water or perform the melt quickly enough that the radiant heat on the coils will be a minimum. Despite the difficulties that levitation melting may provide, it is probably a more reliable method of producing drops of molten steel.

Preparing to melt FLiBe

FLiBe is a toxic salt. Both components, LiF and BeF_2 are toxic, but BeF_2 is worse than LiF. BeF_2 is considered an acute health hazard, illness or death can occur within a week if enough of it is inhaled. Beryllium metal or oxide can also cause a long-term illness called chronic beryllium disease. Because of chronic beryllium disease, our laboratory must not only control the acute hazard of inhaling the salt, but also clean up any loose beryllium on surfaces. BeF_2 is not believed to cause chronic beryllium disease, but our beryllium detection methods do not differentiate between BeF_2 and BeO.

When melting FLiNaBe we had controlled any gases that came out of the vacuum chamber and confined the transfer of salts to a small area of the laboratory. The salts were weighed and mixed in an argon-filled glove box. Open work with the Beryllium salts was performed by personnel in filtered respirator masks. Although we worked very hard at reducing the spread of beryllium, we did get some detectable levels on a wooden workbench that needed to be cleaned several times reduce the level of removable beryllium below 0.2 $\mu m/100\ cm^2$. For this work we decided to also control the air flow around the vacuum chamber to minimize the spread of beryllium salts to the rest of the laboratory. We obtained a metal downdraft table that we plan to attach to our HEPA filtered vent system. The HEPA filter on this vent is a bag-in bag-out design that reduces exposure of the lab to Beryllium.

A test plan is required in our laboratory to test materials such as FLiBe because this beryllium salt is not included in a standard operating procedure for our laboratory. Our laboratory is a beryllium facility and is tested for loose beryllium after experiments are performed that include beryllium. A preliminary test plan was written to encompass future testing the salt and ferritic steel systems. This test plan includes a preliminary test of a KF/LiF combination salt that would have a melting point that is similar to FLiBe, but does not include the beryllium salt. Once we develop a method to reliably drip molten steel, the KF/LiF test will determine if there is an explosive hazard when the molten steel drips into the FLiBe. One would expect some vaporization from the FLiBe, but it may not be high enough to cause over pressurization of the vacuum system. This test will be run with shutters in front of the vacuum windows and Lexan explosive shields on the outside of the window.

The larger aluminum oxide crucible and Ta heater can be used to melt approximately 5 grams of FLiBe. To obtain temperatures of 500-700 ºC probably requires about 300 Adc. The test stand will include a residual gas analyzer to measure the gasses produced when the steel is dropped. After the drop falls into the salt bath, a wire basket inside of the crucible would be lifted out of the salt, removing the steel sample before the salt is cooled. The sample can be analyzed to determine changes chemically.

The vacuum chamber would have to be enlarged to include both heaters, one to melt the steel, and one to melt the salt. This could also take place in the e-beam chamber called EBTS where we test solid beryllium samples. We normally clean up EBTS by blasting the inside surface with CO_2 pellets. Either way, clean up after the test must be good enough to reduce the removable beryllium down to 0.2 μg/100 cm^2.

Conclusions

Within this project we explored various methods of melting and dropping ferritic steel into molten FLiBe bath. The method we first tried, melting the steel in a crucible with a hole in the bottom, proved difficult because there was not enough mass in the crucible to overcome surface tension in the hole. Inductive levitation melting could be more likely to produce a reliable drop. We have started to invest in better equipment for working with beryllium salts by purchasing a downdraft table to attach to our HEPA-filtered vent. We also suggest that KF/LiF salt be used as an alternative to FLiBe in early experiments.

References

[1] R. L. Kleuh, D. S. Gelles, S. Jitsukawa, A. Kimura, G. R. Odette, B. van der Schaaf, and M. Victoria, Ferritic/Martensitic steels – overview of recent results, *Jour Nucl Mater.* 307-311 (2002) 455-465

[2] S. Jitsukawa, M. Tamura, B. van der Schaaf, R.L.. Klueh, A. Alamo, C. Petersen, M Schirra, P. Spaetig, G. R. Odette, A. A. Tavassoli, K. Shiba, A. Kohyama, and A. Kimura, Development of an extensive database of mechanical and physical properties for reduced-activation martensitic steel F82H, *Jour Nucl. Mat.*,307-311 (2002) 179-186.

[3] G. Gervasini, G. Bonizzoni, A. D. Laine, C. Cepek, E Magnano, and M. Sancrotti, Characterization studies on low activation martensitic steels for fusion reactors, *Vacuum*, 48 (1997) 423-426.

[4] R. D. Mathis Company, P.O. Box 92916, Long Beach, CA 90809-2916.

[5] Ameritherm, Inc., 39 Main Street, Scottsville, NY 14546, http://www.ameritherm.com/overview_levitation.html

[6] Lepel Corporation, 200G Executive Drive, Edgewood, NY 11717, http://www.inductionheating.com/

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