Induction Furnace Testing of the Durability of Prototype Crucibles in a Molten Metal Environment

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Abstract

Engineered ceramic crucibles are commonly used to contain molten metal. Besides high temperature stability, other desired crucible characteristics include thermal shock resistance, minimal reaction with the molten metal and resistance to attack from the base metal oxide formed during melting. When used in an induction furnace, they can be employed as a "semi-permanent" crucible incorporating a dry ram backup and a ceramic cap. This report covers several 250lb single melt crucible tests in an air melt induction furnace. These tests consisted of melting a charge of 17-4PH stainless steel, holding the charge molten for two hours before pouring off the heat and then subsequently sectioning the crucible to review the extent of erosion, penetration and other physical characteristics. Selected temperature readings were made throughout each melt. Chemistry samples were also taken from each heat periodically throughout the hold. The manganese level was observed to affect the rate of chromium loss in a non-linear fashion.

Introduction

This work was performed under a CRADA agreement between the US Department of Energy and a private company which had developed several proprietary crucible formulations and needed an unbiased venue to evaluate them in. This company and the identity of the "commercial" crucible used for comparison will remain anonymous. However, we feel that this work has generic value to the air melt foundries with respect to melt chemistry and all induction melting facilities with respect to thermal management. When used in an induction furnace, they can be employed as a "semi-permanent" crucible incorporating a dry ram backup and a ceramic cap [1, 2]. In such applications, a heat is typically melted and the temperature stabilized followed by pouring. In many cases the crucible is recharged with material for a follow up heat. In our research work, it is more common for us to completely cool the furnace prior to melting the next heat which could occur several days hence. Cracks are typically observed in the ceramic cap and crucible, even after one heat; however, crucibles are not replaced until gross defects are observed. In this application, thermal shock from cycling is, in many cases the cause of eventual crucible failure. In a production environment, the crucible will spend more of its time in contact with molten metal and may not cycle back to room temperature until it needs to be replaced. In this investigation, we report on the observations made on several prototype crucibles during controlled furnace tests.

Experimental Procedures

In order to test the stability of each crucible in a molten metal environment, a charge of 17-4PH stainless steel was melted, held molten for 2 hours, then poured. The charging, melting, holding, sampling and tapping procedures for all of the melts were as follows: The charges were loaded into the furnace, the coil was energized with 20KW power and held for approximately 30min. Subsequently, the power was raised 20KW every 30min until the power setting reached 80KW. This was the highest setting that could be reached using this charge/coil combination. It generally took 1 to 1.5h for the charge to be fully molten and reach the holding temperature (1540C). The charge was maintained at 1540C for 2h based on an immersion thermocouple (type R) which was continuously immersed in the bath. The protection sheath for this thermocouple consisted of a high density alumina tube with a high alumina castable formed around the lower half which increased the wall thickness above ~1/2in. The thermocouple position was changed (lowered) about half way through the hold period to increase the sheath life since the greatest erosion occurred at the melt line. The temperature was cross checked against a dip thermocouple throughout the run. Thermocouples were also located on the outside of the

crucible, on the center of the bottom, 4in up from the bottom, midway up, and 4in from the crucible rim. Pin samples were taken at predetermined times throughout each melt run and buttons were poured just as the heat was being poured off into a mold. A bar-shaped sand mold was used to receive the heat and this bar was cut into sections for remelting. In this way, the same material was used for each melt. Additional virgin material was added to subsequent melts to bring the charge weight up to ~260lb. In addition, Mn and FeCr were added to the last melt to bring the charge chemistry more in line with 17-4PH requirements. A typical run would last 5-6h from power on until pouring. Photo documentation was made of all aspects of the experiments. The used crucibles were sampled by taking ~1.5in bore samples at the melt line and mid radius of the crucible bottom. The bore samples were sectioned diametrically, mounted in clear epoxy and photographed at low magnification to document the general condition of these representative areas. Subsequently, these polished cross sections were reviewed in our SEM to document the extent of dross or metal penetration. Note that the outside portion of the crucible was cut off in many cases to facilitate mounting (this is noted in the figures).

Results

Melt Run Observations

The typical appearance of a melt during a run is shown in Figure 1. In each case the melt surface was generally covered in a base metal oxide (dross). The dross was cleared occasionally to take samples; however, no effort was made to remove all of the oxide except just before the heat was cast. An example of a crucible after casting is shown in Figure 2 which is typical of all the melts. Note that some of the dross was still attached at the melt line. This thick dross "ring" is somewhat different than what would develop during more typical foundry practice of charge, melt, pour, repeat. However, it presents a more aggressive environment to test the crucible under. Table I gives the chemistry of the melts at the end of each 2h run as well as the order of melting. A loss of Cr and Mn was observed. During the third melt the heat was observed to be excessively drossy as compared to the previous two melts. The manganese level was observed to have fallen below 0.1 weight percent at the end of this melt. The manganese loss was accompanied by a significant drop in chrome (a loss of 1.85 weight percent chrome form melts 2-to-3 vs. a loss of only 0.55 weight percent from melts 1-to-2). It is well known that the oxidation potential for the formation of manganese oxide is greater than that for the formation of chrome oxide [3]. It is likely that preferred formation of manganese oxide in the dross or on the charge during melt-in helps to minimize the loss of chrome through oxidation. Apparently, a manganese level greater than 0.09 and perhaps greater than 0.16 weight percent is insufficient to form the Mn-rich dross layer on a melt of 17-4PH. As a result of the third melt, the Cr level had dropped to below the specification minimum, thus a Cr (and Mn) addition was made to the 4th melt once the bath was fully molten. The resulting chemistry was still below spec for 17-4PH, however both Cr and Mn were raised. The addition made should have given levels about midway between the min and max, thus additional losses still occurred.

Figure 1: Example of the typical appearance of the melt is shown above. Note the dross (darker material inside the crucible) that remained on the surfaces of the melts for the majority of each of the tests. The immersion thermocouple used to monitor and control the bath temperature is also evident in the photo.

Figure 2: An example of a test crucible is shown above after poring off the heat. Note the heavy dross left at the melt line.

Table I. Order of testing and heat chemistries following the various crucible tests are given below.

	Chemical analysis results (weight percent)						
ID and melt order	Mo	Cu	Ni	Fe	Mn	Cr	Si
D048 – 1st	0.19	3.22	4.17	75.52	0.31	15.64	0.47
D056 – 2nd	0.19	3.25	4.23	76.34	0.16	15.09	0.30
Commercial – 3rd	0.20	3.31	4.30	77.63	0.09	13.84	0.25
043* - 4th	0.19	3.22	4.20	76.35	0.29	14.78	0.57

**Note that an addition of FeCr and Mn was made to the melt test of crucible 043 once the bath had reached ~1500C. The additions were intended to bring the Mn up to 0.3 and the Cr to 16.5. As one can see, the Mn responded nicely while the Cr level remained lower than expected.*

Macroscopic Observations

All of the crucibles cracked to some extent as a result of the thermal cycle and/or extraction from the coil box (Figure 3). This seems to be normal in our experience. Crucible D056 cracked just above the melt line while melting the charge which is unusual. Crucible 043 cracked on the bottom during the run and a small amount of molten metal leaked into the dry ram. This may have been due to unevenness in the dry ram below the crucible.

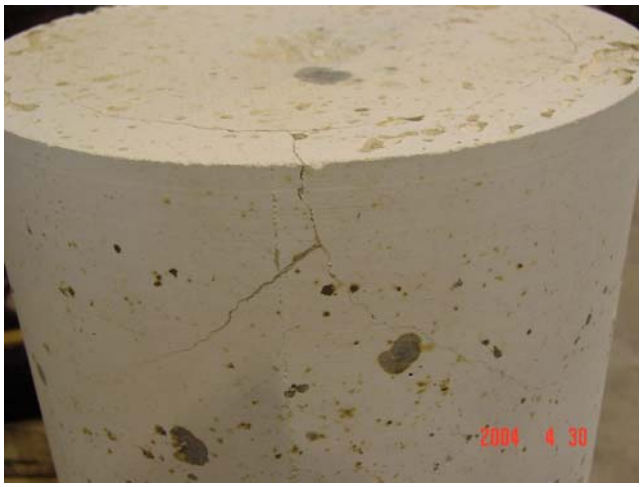

Figure 3: An example of conditions typically observed on many crucibles after testing is shown above (note the cracks). The dark spots were simply "discoloration" from an unknown source and not evidence of metal penetration.

Photomacrographs of cross sections through the crucible bore samples are shown in Figures 4-7. In general, all but the commercial crucible showed obvious erosion at the melt line. It should be noted, however, that the commercial crucible was the third in the melt order and the dross buildup during this melt was very heavy. Perhaps the dross could have formed a heavy, viscous layer at the melt line where the heavy erosion was observed on the other crucibles.

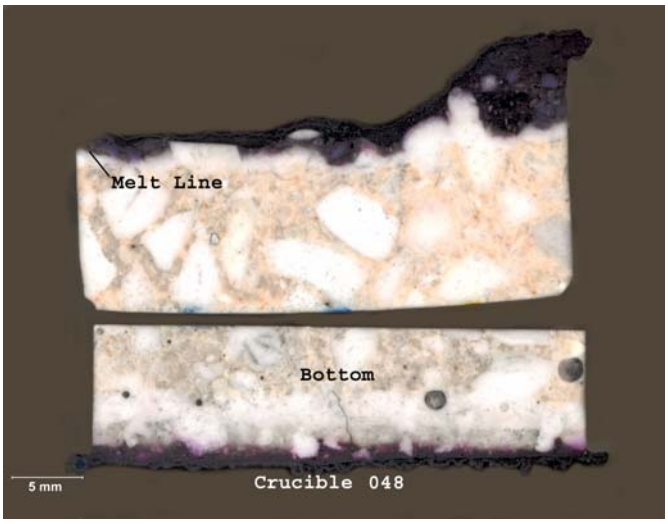

Figure 4: Photomacrograph of the core sample cross sections are shown above. Clear evidence of erosion of the crucible was observed in the sample from the melt line. Note that the back side of the bottom sample had been cut off to facilitate mounting.

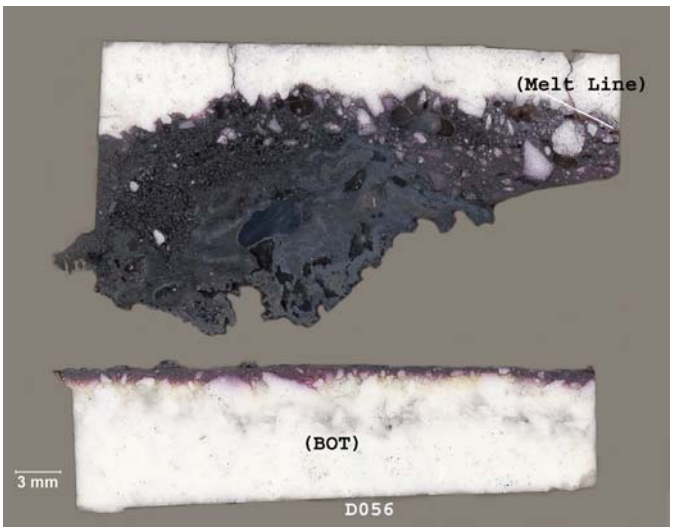

Figure 5: Photomacrograph of the core sample cross sections are shown above. Clear evidence of erosion of the crucible was observed in the sample from the melt line. Note that the back side of both samples had been cut off to facilitate mounting.

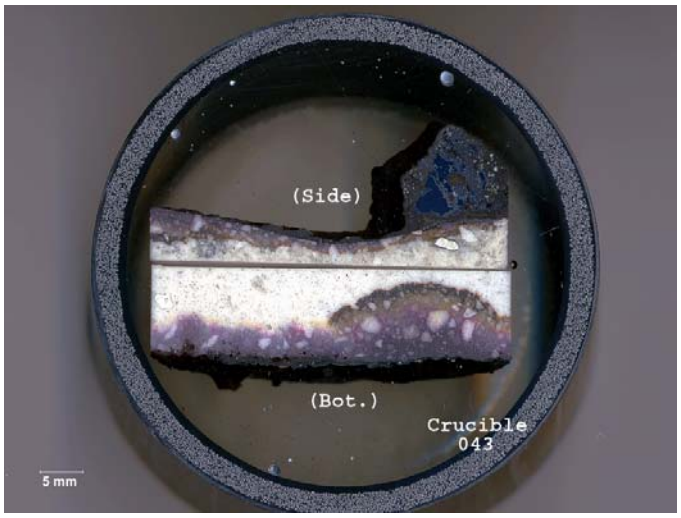

Figure 6: Photomacrograph of the core sample cross sections are shown above. Clear evidence of erosion of the crucible was observed in the sample from the melt line and heavy penetration was observed on the bottom sample. Note that the back side of both samples had been cut off to facilitate mounting.

Figure 7: Photomacrograph of the core sample cross sections are shown above. No evidence of erosion of the crucible was observed. Note that the back side of the bottom sample had been cut off to facilitate mounting.

Microscopic observations

The first test crucible (048) consisted of a calcia-magnesia matrix with an alumina aggregate. While there was little evidence of chrome penetration at the melt line (via the dross which was rich in Cr, Mn and Si) there was significant erosion as evidenced in the macro view (Figure 4). There was a slight deposit of Cr and Si on the cross section through the crucible bottom (Figure 8) indicating only a minor interaction between the crucible and molten metal.

The second test crucible (056) consisted of a magnesia-alumina matrix with an alumina aggregate. Chrome, manganese and silicon were observed to penetrate 2mm or more at the melt line (via the dross which was rich in Cr, Mn and Si) while there was less erosion evident in the macro view of this crucible than crucible 048 (Figure 5). The metal penetration into the bottom of crucible 056 measured about 0.6mm and consisted of chrome and manganese (Figure 9).

The third crucible tested was a commercially available unit. This crucible consisted of a silica-magnesia matrix with an alumina aggregate. It should be noted that the alumina aggregate appeared to be quite a bit finer on the inside (metal side) of this crucible. There was a distinct lack of penetration or erosion at the melt line (Figure 7) and with the exception of some chrome rich drossy material that was adherent, the bottom crucible section remained untouched by the molten metal (Figure 10).

The third test crucible (043), which was 4th in the melt sequence, consisted of an alumina-silica matrix with an alumina aggregate. Gross erosion was observed at the melt line (Figure 6) although the penetration into the crucible at this location was only about 1mm (apparently, once the ceramic matrix was penetrated it was easily eroded away). The metal penetration into the bottom of crucible 043 measured about 5-6mm and consisted of chrome and manganese (Figure 11). This crucible was by far the poorest performer of all those tested. A very nice example of manganese oxide overlaying chrome oxide was found on a cross section of this crucible (Figure 12) giving further support to the idea that a certain manganese level protects the chrome from oxidation during melt processing preventing excess chrome losses.

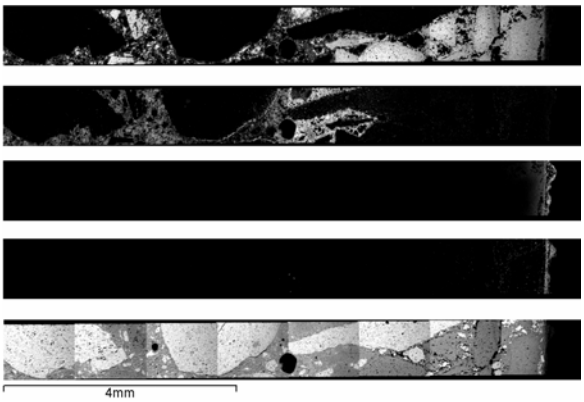

Figure 8: SEM EDS maps of crucible 048 bottom cross section are shown above. The melt/crucible interface is on the right and the images are of Mg, Ca, Cr, Si, and Al from top to bottom.

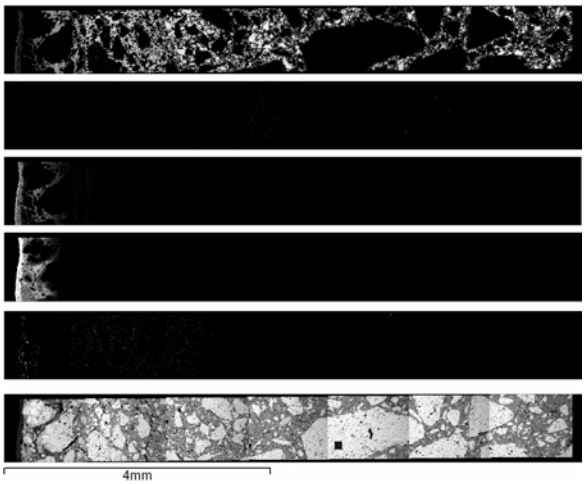

Figure 9: SEM EDS maps of crucible 056 bottom cross section are shown above. The melt/crucible interface is on the left and the images are of Mg, Ca, Mn, Cr, Si, and Al from top to bottom.

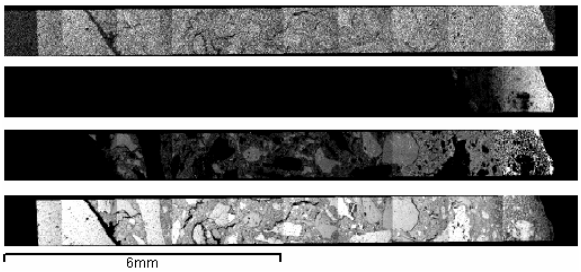

Figure 10: SEM EDS maps of the commercial crucible bottom cross section are shown above. The melt/crucible interface is on the right and the images are of Mg, Cr, Si, and Al from top to bottom.

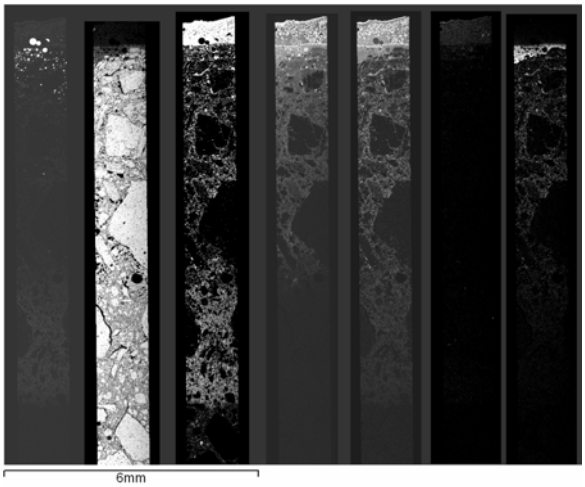

Figure 11: SEM EDS maps of crucible 043 bottom cross section are shown above. The melt/crucible interface is on the top and the images are of Fe, Al, Si, Cr, Mn Ca, and Mg, from left to right.

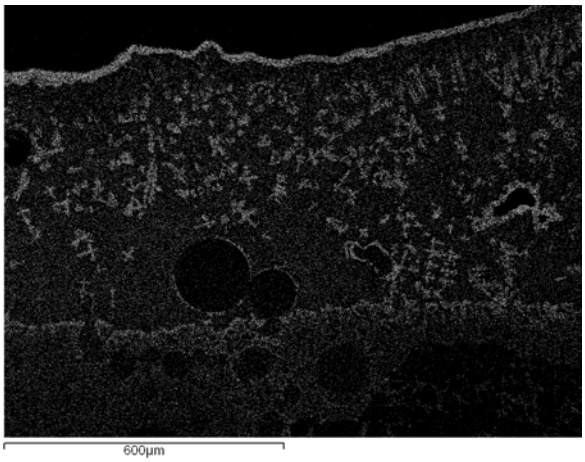

Figure 12: Higher magnification SEM EDS map of Mn on the crucible 043 bottom cross section is shown above. Note how the Mn forms an outer layer (top) on the dross that is formed.

Temperature Measurements

The thermocouple data from each of the melt tests are shown in Figures 13-16. The temperatures on the outside of the crucible ranged from about 800C at the upper wall to over 1300C on the crucible bottom. Note that the upper thermocouple was above the melt line. Considering that the average metal temperature during the melt was 1540C, the temperature gradient through the crucible wall was observed to be on the order of 300-500C. In general, the immersion thermocouple was quite useful for maintaining the bath temperature. In each of the plots, one can see how the immersion thermocouple responded to manual changes in KW settings. Dip readings were also taken at various times during the runs as a secondary check of the temperature. In most cases, these readings were lower than the immersion readings and as such are only reported for crucible 048. This disparity in temperature readings was likely due to a buildup of metal and dross on the tip of probes (dip probes were reused several times) as well as the location of the reading (always near the bath surface, as opposed to an inch or two submersed). During the initial test of crucible 048, a gradual fall of the bath temperature was observed. Meanwhile, the dip readings were beginning to read above that of the immersion readings. Further investigation revealed that the immersion thermocouple junction was just at the bath surface and starting to fall below due to dross formation. Immersing the probe further into the bath corrected this problem, giving a truer reading. A corrected version of the immersion readings is also given in the plot. The average temperature over each of the runs was about 1540C (~2800F).

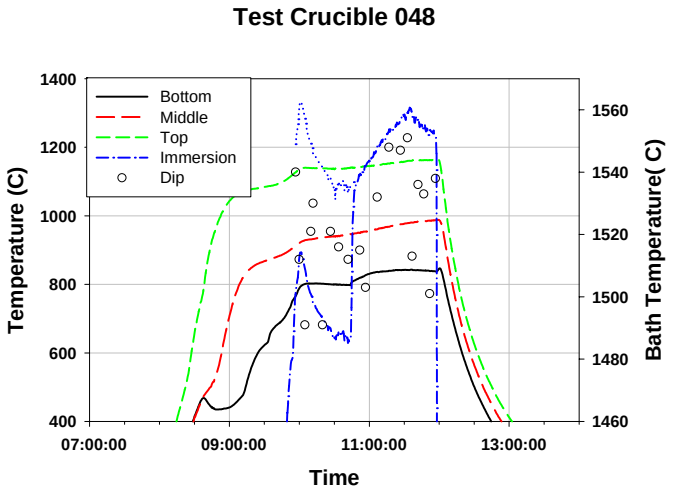

Figure 13: The crucible outside temperature (left axis) and bath temperature (right axis) are shown versus time in the plot above. Note how the dip temperature reading initially measured above the immersion reading. The immersion thermocouple was lowered further into the bath at ~10:45 which gave a truer reading of the bath. The approximate true reading during this time is shown as a dotted line.

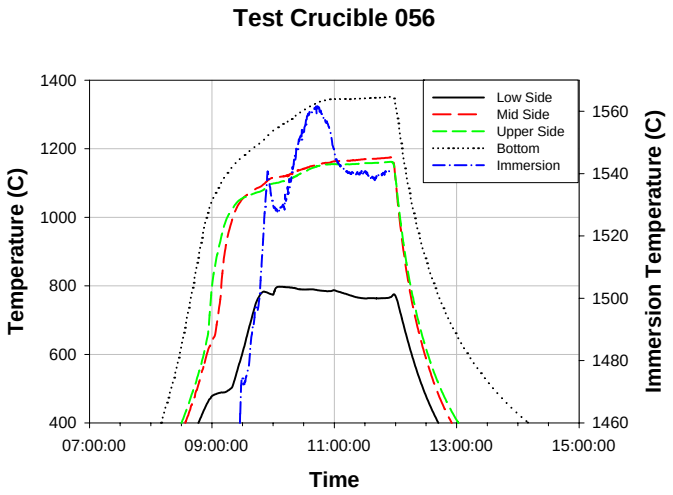

Figure 14: The crucible outside temperature (left axis) and bath temperature (right axis) are shown versus time in the plot shown above.

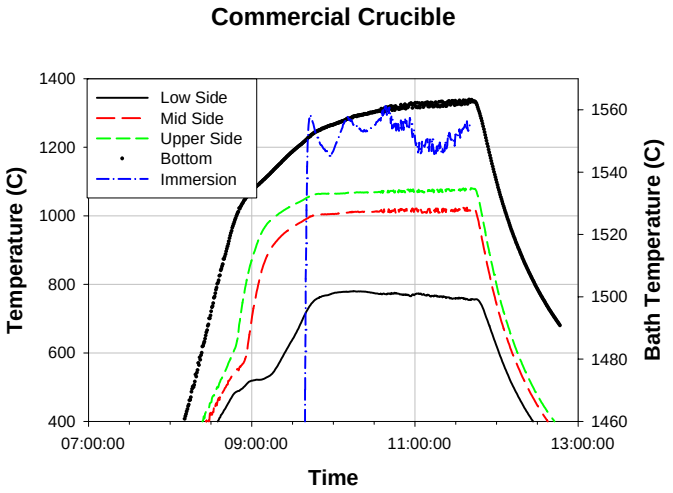

Figure 15: The crucible outside temperature (left axis) and bath temperature (right axis) are shown versus time in the plot shown above.

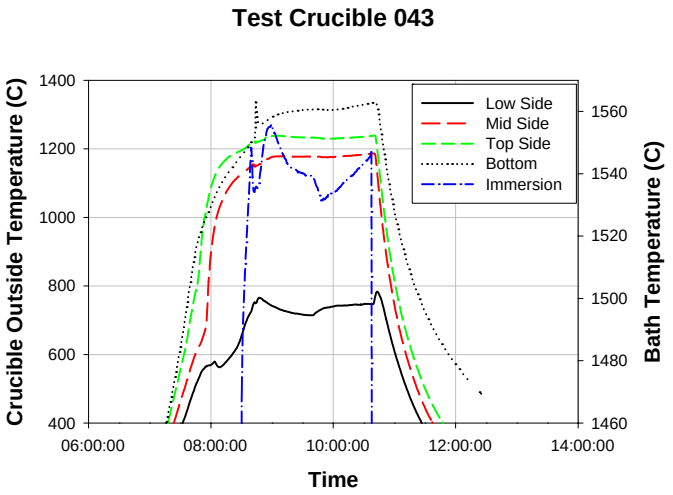

Figure 16: The crucible outside temperature (left axis) and bath temperature (right axis) are shown versus time in the plot shown above.

In-Process Melt Chemistry

During melt processing of each of the heats, melt samples were taken at melt hold times of 0, 15, 30, 60, and 120 minutes. The results of the chrome analysis are shown in Figure 17. Melts 1 and 2 are grouped together and a linear regression line fits the data well. Melts 3 and 4 are plotted separately. A linear regression line has been fit to the melt 3 data. It is clear from the plot that melts 3 and 4 are distinctly different from the combined results of melts 1 and 2. Ferro-chrome and manganese additions were added at the beginning of melt 4 which explains the different results for this heat. The evidence from this plot suggests that something was different during the third melt heat up. The only other

significant change in chemistry we observed during the four melts was the change in manganese (Table I). The manganese results are plotted in the same manner as the chrome results in Figure 18. The loss of manganese appears to be more like an exponential decay. Also, unlike the results from the chrome analysis, melts 1 and 2 are easily distinguished by the large pre-hold loss of manganese during the 1 to 1.5h melt-in period. From melts 2 to 3 the manganese level drops from 0.15 to 0.1 weight percent. Also, the manganese loss during the third melt appears to be minimal. Our dross observations during the third melt were that this dross was excessively heavy. The chrome level dropped precipitously during the heat up of the third melt while the chrome loss during melting was at the same rate as before (i.e., the two regression lines are parallel). Thus, it is our contention that a minimum amount of manganese is desirable to reduce the chrome losses during the heat up stage of melting. This minimum Mn level is greater than 0.1 and may need to be as high as 0.3 weight percent, the level of Mn at the end of the first melt.

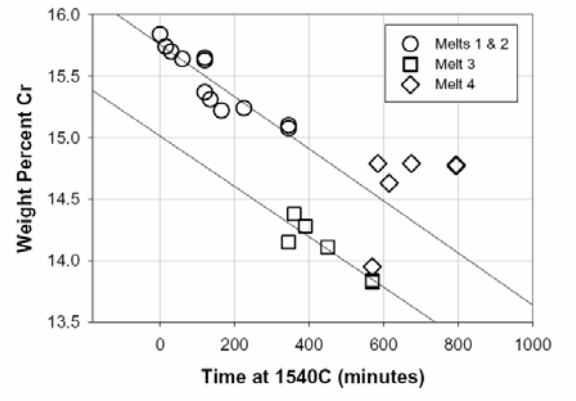

Figure 17: Weight percent chrome is plotted above as a function of hold time at 1540C. Melts 1 and 2 are grouped together while 3 and 4 are plotted separate. Two regression lines are plotted: one for melts 1 and 2, a second for melt 3.

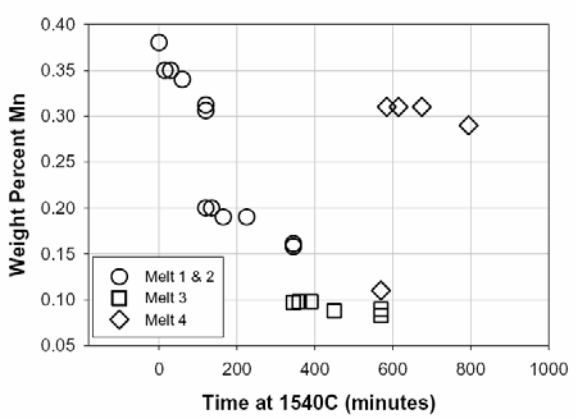

Figure 18: Weight percent manganese is plotted above as a function of hold time at 1540C. Melts 1 and 2 are grouped together while 3 and 4 are plotted separate. Two regression lines are plotted: one for melts 1 and 2, a second for melt 3 as per Figure 17.

Acknowledgements

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References

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