# EFFECT OF WATERING AND NON-WATERING COOLING RATES ON THE MECHANICAL PROPERTIES OF AN ALUMINUM SMELTER'S POTSHELL

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Keywords: Aluminum Reduction Potshell, Mechanical Test, Cooling Rate, Shutdown Pot, Microstructure

#### Abstract

The cooling time of a shutdown aluminum reduction pot depends on the cooling method. Free-convection cooling (non-watering) takes about five to nine days depending on the surrounding environment while watering the pot shortens the cooling time to less than one day. Application of watering cooling rates on the pot from a high temperature can have consequences on the final mechanical property of the potshell. Facilitating a design of a cooling system which optimizes the cooling rate of a shutdown pot without deteriorating the desired mechanical properties of the potshell is required.

This study focuses on the effect of different cooling rates on the microstructure and mechanical properties of the potshell material. To this effect, samples of potshell material from a shutdown pot were collected and ASTM standard specimens for mechanical and microstructural examinations were machined. Specimens were heated up to a maximum temperature of 500°C and then cooled at various rates ranging from that of free convection cooling to water quenching cooling. Tensile and micro-hardness tests were carried out to examine the effect of these cooling rates on the mechanical properties of the material. Microstructural analysis was carried out to study the microstructural response of the material to the various cooling techniques. The variation of the thermal and thermo gravimetric properties of the potshell material with temperature has also been measured according to ASTM E1461 Test Method.

# Introduction

Modern aluminum reduction pots are lined with rectangular steel pot-shells. As fabrication of this shell is expensive, when the electrolytic cell reaches the end of its useful life, the potshell is usually retained and relined. This necessitates the careful handling of the potshell during and after the life span of the pot.

However, there is usually a delay between the shutdown (electric power cut out) of a failed pot and the start of the actual rebuilding process. The bath and metal would have to be tapped and the anode would have to be removed from the pot in the pot room. This takes about 20 hours. Thereafter, the pot is transferred to the de-lining area and left to cool down to a temperature at which the pitting worker can work on it. This cooling stage may take about 5-7 days if left to cool down in a free convection atmosphere. Reduction of this time is strongly desirable by the aluminum industry to reduce the cost per product in the aluminum smelter.

Watering of the pot is a well-known means of substantially reducing this down time but this process could have a deteriorating effect on the microstructure and mechanical properties of the potshell depending on the thermal history and metallurgy of this material. Also, this method has a negative impact on the environment. Many investigators have extensively studied the effect of different cooling rates on the overall mechanical properties of steel materials [1-6]. However, the flexibility of steel to produce a wide range of physical and mechanical properties by the variation of the composition makes the outcomes from such studies not fully applicable for all steel materials [2]. No study has come across which has considered the pot-shell material that is characterized by chemical composition as shown in table 1. This necessitates physical experimentation where the resulting mechanical properties can be related to the microstructure as validation.

The primary aim of this work is to study the thermal behavior of the potshell made out of the material shown in table 1 and investigate the effect of various cooling rate parameters on the microstructure and mechanical properties of this material. This is carried in view of building an efficient and safe cooling system that will reduce the layoff time of the failed pot to be de-lined.

Table 1: Chemical Composition % of the Ladle Analysis on the Pot shell Material

Nominal thickness =< 40(mm) C max = 0.21									
Nominal thickness = $< 40(\text{mm}) \text{ CEV max} = 0.40$									
С	Mn	Р	S	N	Cu	CEV			
max 0.22	max 1.5	max 0.04	max 0.04	max 0.012	max 0.55	max 0.42			

## **Experimental: Materials and Methods**

Sample Preparation and Cooling Rates Experiment

ASTM standard specimens were machined for the mechanical test. Figure 1 shows the mechanical test specimen schematic. Same specimens are used for all cooling rate considered.



Figure 1. ASTM standard samples for the mechanical test The samples were separated into four categories: Two of which were heated up in a furnace to a maximum temperature of  $400^{\circ}$ C each at heating rates of 5°C /min and 15°C /min with no waiting time. This procedure was repeated for the other 2 categories but with a maximum temperature of 500°C. The cooling was carried out in three different modes: free convection, forced convection (fan with variable speed) and quenching in water. K-type thermocouples were fixed on the samples throughout the whole test period for accurate measurements of the temperature history at sampling rate of 1 Hz during heating and 10 Hz during the cooling processes respectively.

A tensile test was then carried out at room temperature on an Instron-5900 series Tensile Testing Equipment. Specimens for microstructural investigation were cut out from the broken tensile test specimens. The cut samples were then ground and polished to a grade of  $0.04\mu$ m. Nital, a nitric acid-methanol mixture was used to etch the specimens to reveal the microstructure features. Scanning Electron Microscope (SEM) images were taken for each sample. The samples were then transferred to the micro-hardness indenter and the hardness of the pearlite grains was recorded. Figure 2 shows the setup for heating up the samples with thermocouples fixed at both ends.



Figure 2. Setup for heating up the steel samples with thermocouples fixed at both ends

## Thermal and Thermo-gravimetric Properties

The thermal diffusivity and specific heat capacity of the sample was recorded as a function of temperature. The tests were performed in a nitrogen atmosphere according to the specifications of the ASTM E1461 Test Method. The instrument used for the tests was a DLF-1600, able to perform thermal diffusivity measurements on reference material with an expanded uncertainty of  $\pm 2.1$  % for 95 % confidence level. The specific heat capacity results were obtained up to 600°C only due to the behavior of the material.

Thermal conductivity values were calculated from the thermal diffusivity, specific heat capacity results, and room temperature density measurements assuming the material tested is homogenous.

Thermo gravimetric analysis was carried out on a Perkin Elmer TGA4000. A piece of the material weighing 19.885g was heated inside the equipment, in a pure oxygen environment, at a heating rate of  $5^{\circ}$ C /min to 500°C and held at that temperature for 1hr.

## **Results and Discussion**

#### Free and Forced Convection Cooling

The temperature histories of samples during the free and forced convection cooling (air) at different speeds are presented in Fig 3.The diversity of the plots indicate the cooling curve dependency on the cooling-fluid's flow rate and hence the convection heat transfer mode. Reynolds number indicates the flow regime of the cooling fluid and is calculated as:

$$Re = \rho V L / \mu$$
 (1)

Where  $\rho$  is the air density, V is the fluid velocity, L is the sample width, and  $\mu$  is the fluid dynamic viscosity.

When the specimen was left in a free convection environment, it took about 36 minutes to cool down to room temperature while, it took about 14 minutes during the forced convection cooling with Re=830 to reach that same temperature. On the other hand, the cooling time was just about 9secs when the specimen was quenched in water.

The resulting stress-strain curves from the tensile test of the control, free convection cooled, and forced convection cooled specimens are presented in Fig 4. The control specimen was machined from the potshell without prior heat treatment. An ultimate tensile strength of 427 MPa, yield strength of 279 MPa, and percentage elongation of 43% were recorded for the control sample. This is consistent with the data from the suppliers of the steel material (yield strength of 275 MPa and ultimate strength of 400-560 MPa) indicating that the lifetime operation and cooling of the pot had no significant effect on the strength of the potshell cut out samples that had been used in our experiments. The test was repeated two times, and the uncertainty is calculated based on the error in the specimen machining, handling from the furnace to the cooling environment, and reading equipments. The total uncertainty was estimated to be 7.5% with 95% confidence. The effect of free convection cooling increased the yield strength by 13%; however, it showed insignificant effect on the ultimate strength and ductility as shown Fig 4.



Figure 3. Cooling curve of the steel sample at various cooling flow rates of air during free and forced convection



Figure 4. Stress versus strain curve for steel sample after cooling using different air flow rates.

## Water-Cooling

The effect of the heating and cooling rates on the yield and ultimate strength of the quenched specimens is presented in Fig 5. As seen in Fig 5, there is no significant variation in both the yield and ultimate strength of the control sample and specimens heated at any rate (5°C/min or 15°C/min) to 400°C and then quenched. These values showed a variation of less than 1% in both the yield and tensile strength from the control sample. These are within the safe limit according to the material specifications document. On the other hand, the sample heated at an average rate of 5°C/min to 500°C and then quenched in water showed a significant reduction in the yield and ultimate strength values. The yield strength of this sample reduced to 220MPa and the tensile strength reduced to 370MPa. These values are lower than the benchmark values of the original material. Upon heating the same sample at a faster average rate of 15°C/min before water quenching, the yield and tensile strength values recorded were 290Mpa and 440Mpa respectively. Overall there was no significant change in the elastic modulus of all specimens.

These results show slow and fast cooling have a negligible effect on the yield and tensile properties of the material if the steel material is heated to a maximum temperature of 400°C at 5°C/min. This can be attributed to the low recrystallization volume fraction of low alloy steel materials below 500°C [7-9]. Quingge et al [7] reported that below 500°C the crystallization volume fraction of ferrite is about zero hence the only recovery process that takes place in this temperature region involves both the annihilation of dislocations and their organization into lower energy configurations.

On the other hand, fast cooling have a significant effect on the strength of the material heated to a maximum temperature of 500°C at 15°C/min. This can be attributed to the recovery and recrystallization of ferrite [7-9], spheroidization of cementite [8], coarsening of carbide and, microstructural deterioration which could all occur at this temperature range.



Figure 5. Effect of Heating Rate & Cooling Technique on the Yield &Ultimate Strength of the Water-Cooled Steel Sample

Metallurgical Examination of Water Quenched Specimens

The SEM images of the samples are shown in figure 6. The dark regions are ferrites while the regions with white and dark lamellar shaped structure are pearlite [10]. The size and density of the pearlite phase which is a mixture of alternate strips of ferrite in a cementite matrix is seen to be more in the control specimen compared to that of the specimen heated to a maximum of  $500^{\circ}$ C at a rate of  $5^{\circ}$ C/min and then water quenched. This is not the case for the samples cooled from a maximum temperature of  $400^{\circ}$ C and that cooled from  $500^{\circ}$ C but heated up to that temperature at a rate of  $15^{\circ}$ C/min

This confirms that the recovery of ferrite occurs in the sample heated to 500°C at a rate of 5°C /min and then water quenched. The resulting microstructure of most heat treated metals is a matrix of ferrite with the exact amount of ferrite depending on the treatment approach taken. Generally, the recrystallization process is related to the steel chemical composition, specimen holding time, and heating rate etc. [7, 8]. During recrystallization, the ferrite and carbides will nucleate, leading to grain coarsening. The outcome is a change of constituents of ferrite and pearlite phases of the steel material; the grains of ferrite grow and their coagulation occurs while pearlite is diminished.

Thus, the microstructure is mainly composed of ferrite matrix and small amount of colonized carbides. Grain refinement which is the opposite of grain coarsening is one of the important strengthening mechanisms in materials [11]. The finer the grains, the larger the area of grain boundaries that impedes dislocation motion. Also, the hard and strong nature of the pearlite grains as compared to ferrite means pearlite depletion also acts in reducing the bulk hardness and strength of the material. As the ferrites coarsening, the lamellar of the pearlites depletes which leads to reduction in strength and hardness of the pearlite grains and hence the steel sample in whole.



Figure 6. Microstructure of (a) Control Sample (No Heat Treatment) 5000X Magnification (b) Water-cooled from 400°C (heating rate of 5°C/min) 5000X Magnification (c) Water-cooled from 500°C (heating rate of 15°C/min) 5000X Magnification (d) Water-cooled from 500°C (heating rate of 5°C/min) 5000 Magnification

Apparently, the effect of grain coarsening was depicted in the sample heated to 500°C at a rate of 5°C/min and then water quenched. However, this behavior was not observed in the other sample heated to 500°C. This can be attributed to the relatively fast rate at which the sample was heated up and the lack of holding time in the procedure. At that rate of heating, the above mentioned mechanisms are not given enough time to act. As such, the grain sizes are not affected hence the negligible microstructural and mechanical property deviations observed.

The micro-hardness of the pearlite grains as a function of water quenched specimen's temperature and heating rate is presented in table 2. The results in table 2 are measured at three random positions on the specimen surface. The average results of these three positions imply a similar trend to that has been seen for the mechanical strength that is shown in Fig 5. The hardness of the pearlite grains for the samples heated to a maximum temperature of 400°C and then water quenched showed no significant variation from the control sample. Also the sample heated up to 500°C at a rate of 15°C/min and then water quenched shows no significant variation in the hardness of the pearlite grains. However, the sample heated at a rate of 5°C/min to 500°C and then water quenched showed a significant drop in the Vickers Hardness value. Again, the microstructural changes due to recrystallization can be attributed to these trends.

	Position1 (HV)	Position2 (HV)	Position3 (HV)	Average (HV)
Control Sample	304.10	311.50	330.10	315.23
400°C, 5°C/min	334.11	352.80	338.60	341.84
400°C, 15°C/min	332.50	330.75	336.35	333.20
500°C, 5°C/min	284.10	289.70	247.00	273.60
500°C, 15°C/min	330.10	308.70	334.10	324.30

Table 2: Vickers Hardness (HV) of Samples

## Thermal and Thermo-gravimetric Properties

The result of the thermo gravimetric analysis is plotted in Fig 7. The weight percentage of the material increases from room temperature to about 100°C at a somewhat constant rate and remain uniform till about 400°C. At 400°C the weight of the sample then begins to rise again at a faster rate till 500°C and continues to rise as it is held at that temperature. Evidently, the increase in weight of the sample is due to the chemical reaction of the sample with oxygen (oxidation). The result of Fig 7 shows that the oxidation rate increases rapidly beyond 400°C. This result is substantiated by the visual examination of the specimens. The color of the specimens cooled from 500°C was brownish which suggests rust. An image of the broken specimens is presented in Fig 8.

The thermal properties of the potshell as measured are shown in Figs 9 and 10. The interaction between the thermal conductivity and specific heat with varying temperature indicated that the potshell heat transfer rate will be decreased at high temperature. The thermal conductivity variation with temperature becomes nonlinear when the temperature reaches 300°C. This behavior is slightly reduced when the reaches 400°C while the specific heat has almost consistent increase with the temperature. As a result of this interaction the thermal diffusivity in Fig 10 shown consistent linear reduction until the temperature reaches 704°C where it reaches its minimum point then starts to increase. It can be seen from the thermal properties that the potshell material has a slight behavioral change at a temperature range of 300-500°C; however this behavioral change did not affect the potshell material's strength significantly.



Figure 7. Thermo Gravimetric Analysis of pot shell material



Figure 8. Visual Examination of Broken Tensile Test Samples



Figure 9. Specific heat and thermal conductivity of the pot shell material



Figure 10. Thermal diffusivity of the pot shell material

## Conclusion

The effect of cooling rates on the potshell mechanical strength has been studied at various operating conditions. Mechanical and thermal analysis of the potshell samples is presented at different cooling rates using free and forced convection in air and water quenching. Strength measurements showed that the potshell strength was not adversely affected by the life time operation procedures of the pot nor the free convection cooling technique adapted. The effect of the cooling rate on the potshell cut off samples does not only depend on the maximum temperature attained by the specimen but also depends on the thermal history of the specimen in terms of heating rate. The thermal and mechanical results indicated that the potshell behavior changes between 300 and 400°C will not significantly influence the mechanical strength of the potshell material. These results are vital to be used for increasing the cooling rate environment of the potshell and hence reduce the layoff time of the failed pot.

#### Acknowledgements

We would like to acknowledge the Emirates Aluminum (EMAL) for support as sponsor for this research. Also, we would like to acknowledge the reduction service department at EMAL for their sincere collaborations.

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