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Measurement of dissolved hydrogen in Al alloys and calculation of hydrogen removal efficiency of a degassing system

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ABSTRACT

In this paper we reported the diminution pattern of hydrogen contents in molten aluminium alloys during casting on industrial scale, and estimated the efficiency of inline rotary degassing system. The degassing parameters were tried to set constant in nearly 150 heats, so that other factors which govern the degassing efficiency of inline unit could be in-sighted well. In this study we used Leco RHEN-602 hydrogen determinator based on the principle of hot extraction carrier gas method, for the measurement of dissolved hydrogen in molten metal and from these results efficiency of inline rotary degassing unit was calculated. The results obtained were cross checked by observing hydrogen porosity in final castings; it endorsed the values obtained through RHEN-602 analyzer. This study showed that hydrogen removal efficiency of the rotary degassing unit was nearly 40 % for various aluminum alloys.

Keywords: Degassing, hydrogen contents, molten metal, porosity, removal efficiency

1. INTRODUCTION

Hydrogen is the only gas that is appreciably soluble in molten aluminum and its alloys[1-3]. The solubility of hydrogen in aluminum increases rapidly with increasing temperature[1].Hydrogen in the liquid melt may arise from a number of sources i.e. disassociation of atmospheric moisture, products of combustion in gas fired furnaces, dissolved hydrogen in raw materials, moisture contamination of fluxes, incompletely dried furnace refractory and contamination from tools, flux tubes, ladles etc. Liquid aluminium has much larger solubility for hydrogen than solid aluminium, as a result during casting process hydrogen is released from the melt, creating pores in cast products. Although majority of the hydrogen is released, a small percentage may get trapped, creating hydrogen filled voids. This remaining hydrogen gas is the root cause of numerous failure mechanisms in aluminum products. Some authors[4-6] have reported the reduced mechanical properties and lower corrosion resistance due to hydrogen porosity.

The assimilation of hydrogen can be minimized by proper melting techniques[7], whereas dissolved hydrogen could be removed from the melt before casting by means of several techniques[8-12]. Degassing is the most common and effective way of reducing porosity due to hydrogen. Degassing methods utilizes chemically pure and dry gases; the most common are argon and nitrogen. The second one is cheaper and used most often for degassing purpose. Mixtures of reactive gases such as chlorine with nitrogen are also used and aid in cleaning the melt from inclusions[13-14]. During degassing bubbling of argon or nitrogen is done by means of a degassing rotor, which reduces the bubble size and disperses the carrier gas throughout the molten metal bath. As the carrier gas bubbles rise through the mass of molten metal they absorb the dissolved hydrogen and remove it from the melt. The efficiency with which the hydrogen is removed is directly related to the bubble size and gas dispersion within the melt[15-16]. It is a costly process so precise knowledge of the hydrogen content in the melt before and during degassing process is highly desirable. A number of techniques have been emerged to measure the degassing efficiency .i.e. reduced pressure test, electrochemical hydrogen sensor etc. In Pakistan peoples never choose a number of equipments for a similar purpose, for example in the presence of a laboratory type hydrogen analyzer they don't spend money on probe type analyzer. Therefore in this study we have employed the Leco hydrogen determinator (which analyzes solid samples only) for the estimation of degassing efficiency. We calculated the degassing efficiency from the hydrogen contents of holding furnace samples and hydrogen contents at casting station as follow;

$$\text{Efficiency} = (C_{H.Furnace} - C_{Casting\ Station})/C_{H.Furnace}$$

Where $C_{H.Furnace}$ and $C_{Casting\ Station}$ are the respective concentrations of the hydrogen gas before and after the degassing treatment.

2. EXPERIMENTAL

2.1 *Sampling and Sample Preparation*

Sampling of molten metal was done using a cylindrical copper mold to minimize the formation of voids. Sampling spoon and the mold was preheated before sampling of molten metal. After collection the sample was cooled instantly with cold

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water. Solid samples were machined to get a uniform dimension of 10 x 24 mm. Sample size & weight, speed of lathe machine and other parameters were followed as described in Leco application notes[17-18]. After machining, the samples were washed with AR grade acetone and dried in warm air. All the prepared samples were handled with clean tweezers and analyzed quickly to minimize the surface contaminations.

Two samples from holding furnace before the melt moved into the degassing unit and two samples from casting launder after the degassing process were collected for the estimation of removal efficiency.

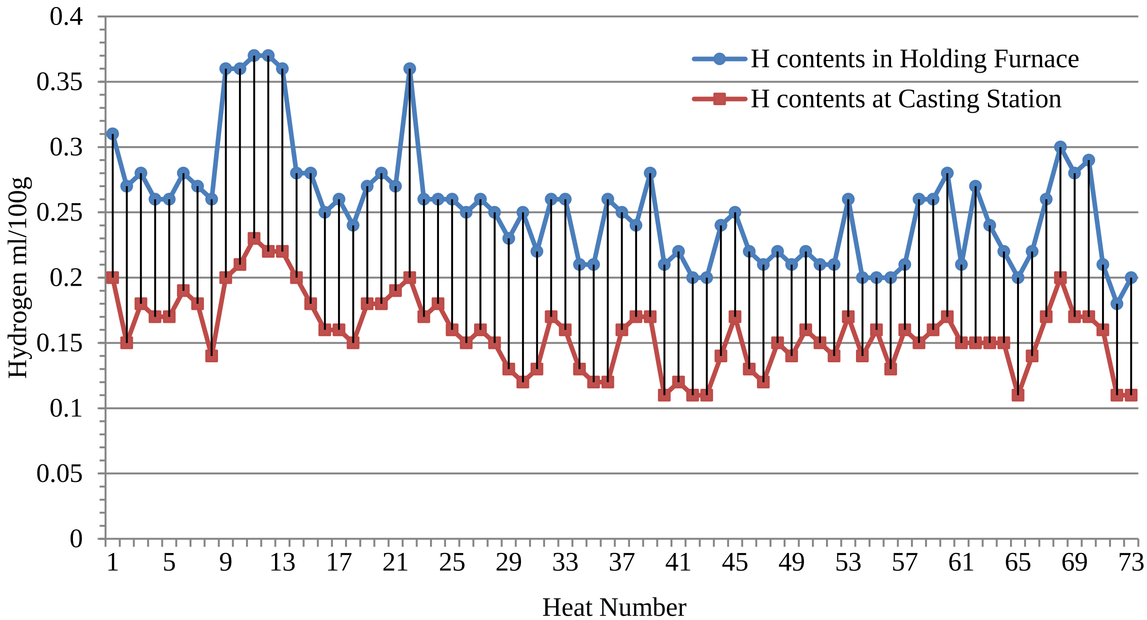

Fig-1: Hydrogen diminution pattern in 6xxx

2.2 *Method and Equipment*

Hydrogen gas continents in Al alloys samples were determined according to the standard procedures described in literature[17-19]. The equipment used was Leco hydrogen determinator RHEN-602.The working principle of this machine is based on the fusion of a sample in graphite crucible at a temperature above the melting point of that substance in an inert gas such as argon. Subsequently the thermal conductivity technique is utilized for the measurement of evolved hydrogen. The equipment was calibrated by reference standard materials provided by Leco, and drift was checked and corrected at regular intervals. The read out from equipment was in ppm unit and in % value by weight; the results were converted into ml/100g units purposefully for comparison with available literature.

2.3 *Macro etching of cast samples*

Hydrogen porosity test was conducted on casting logs of each heat. A 20 mm slice of the cast log along the cross section was taken and soaked in 10% NaOH solution for about 10 minutes. After washing the sample with tape water it was immersed in 10% HNO_3 for 5 minutes and then washed with water and dried in hot air. All the etched samples were checked for hydrogen porosity/ pin holes with magnifying glass and under the stereomicroscope.

3. RESULTS AND DISCUSSION

The results of molten metal samples before and after the degassing system have been plotted in Figure 1 to Figure 6. The upper curve having circle dots represents the hydrogen value as H_2 ml/100g Al in samples taken from holding furnace just before feeding the molten metal to rotary degassing unit. The lower curve having square dots represents the hydrogen concentration in molten metal after the degassing process. The drop line which connects the upper and lower points, represents the actual decrease as ml/100 g, from this value we have calculated the % efficiency of the degassing unit.

3.1 *Hydrogen contents of AA 6xxx*

Under 6xxx series 6011, 6061 and 6063 alloys were studied, hydrogen contents in the holding furnace ranged 0.18 minimum to 0.37 maximum. The average value in 73 heats was found 0.25 ml/100 g. It is notable that most of the high values in holding furnace samples are on the left side of the graph. Some of the values even crossed the 0.35 line; heat number 9 to 13 and heat number 22 showed maximum H_2 values. While investigating it was revealed that higher moisture levels of atmosphere around the holding furnace significantly contributed and raised the hydrogen contents of the molten metal. The results of samples collected just before casting demonstrated the actual decrease in dissolved hydrogen contents while passing through the degassing unit. Hydrogen concentration in liquid metal after degassing treatment was found as low as 0.11 ml/100 g. The average hydrogen value in casting samples of 6xxx remained at 0.16 ml/100 g. The removal efficiency of rotary degassing unit ranged from a minimum 20% to 53.8% maximum in alloys of 6xxx series (Fig-6).

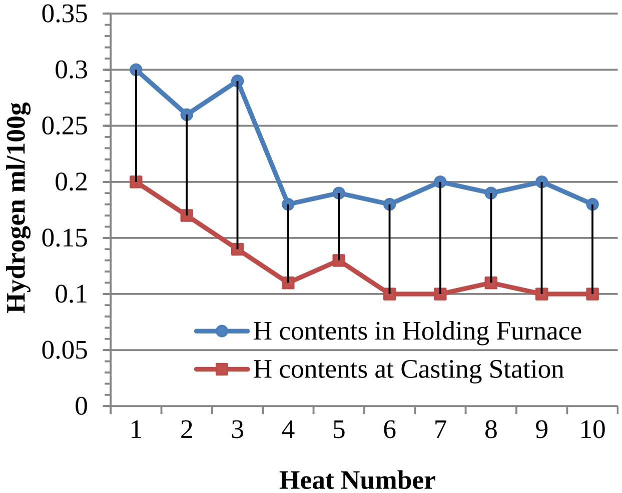

Fig-2: Hydrogen diminution pattern in 5xxx

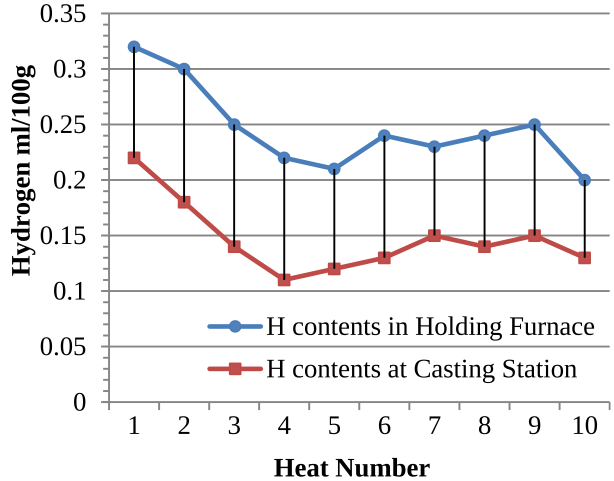

Fig-3: Hydrogen diminution pattern in 3xxx

3.2 *Hydrogen contents of AA 5xxx*

The main alloys of 5xxx series casted during this study were 5050, 5052 and 5083. Hydrogen concentration varied in holding furnace samples from 0.18 – 0.30 ml/100g with an average value of 0.22 ml/100 g. It was observed that whenever greater quantities of scrap metal was used in the manufacturing of alloy, greater hydrogen contents were found in the holding furnace samples i.e. melting number 1, 2 and 3 utilized 20-30% in-house scrape (Fig. 2). Molten metal samples collected after degassing ranged 0.10 -0.20 with an average of 0.13 ml/100g. The maximum decrease was observed in melting number 3 and it was 51.7% as shown in Fig-6.

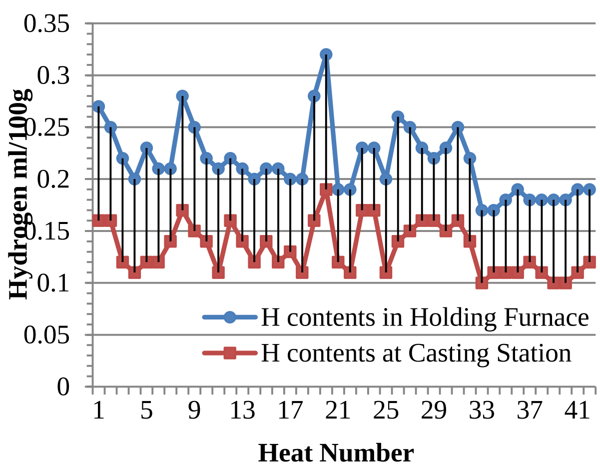

Fig-4: Hydrogen diminution pattern in 2xxx

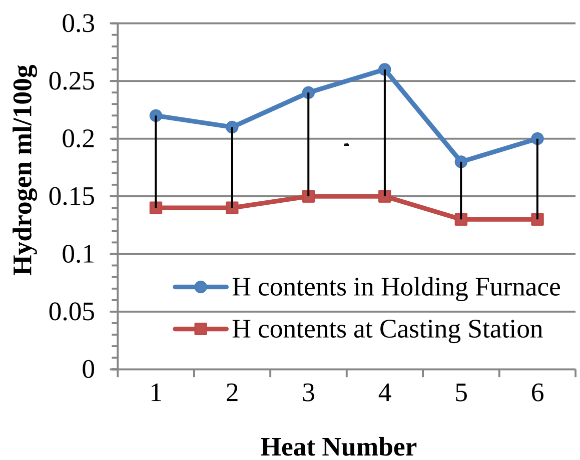

Fig-5: Hydrogen diminution pattern in 1xxx

3.3 *Hydrogen contents of AA 3xxx*

The graph of 3xxx series looks like a perfect ladder (Fig. 3), average decrease in hydrogen value was found 0.1 ml/100g while moving from holding furnace to casting station through rotary degassing unit. The removal efficiency varied in the range of 31.3% minimum to 50% maximum (Fig. 6).Hydrogen contents at casting station were below 0.15 ml/100g in most of the heats, which is a minimum requirement for good quality castings.

3.4 *Hydrogen contents of AA 2xxx*

The 2xxx series under study comprised of 2014, 2018, 2024, 2036, 2050, 2219 and 2618 alloys. Hydrogen concentration in holding furnace ranged 0.17 -0.32 with an average of 0.22 ml/100 g (Fig-4). The highest values were observed in the rainy season when the moisture in air was high. After degassing average H_2 value obtained was 0.13 with a minimum 0.10 and a maximum 0.19 ml/100g in samples collected from casting launder just before casting. Average removal efficiency of degassing unit was lingered at 38.6% (Fig-6).

3.5 *Hydrogen contents of AA 1xxx*

In 1xxx series 1050 alloy was casted during this study; from fig. 5 it is noticeable that there is no elevated reading in holding furnace samples, H_2 values remained below the 0.30 barrier. But on the other hand neither of the casting samples came down to 0.1 ml/100g level, in fact all samples after degassing hang around 0.15 ml/100g value. We observed 35.4% average hydrogen removal efficiency of the degassing unit for 1xxx (Fig-6).

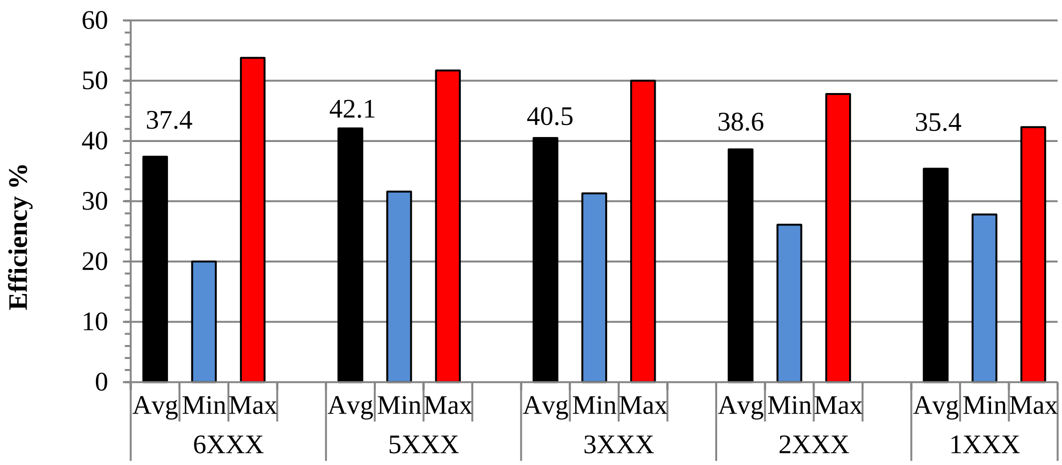

Fig-6: Hydrogen removal efficiency of inline rotary degassing system for various Al alloys

3.6 *Hydrogen removal efficiency*

The hydrogen removal efficiency of inline rotary degassing unit has been plotted in Fig. 6, which shows the minimum, maximum and average efficiency in various series of Al alloys under study. The removal efficiency ranged 20-53.8% with an average value of 38.4% in various aluminium alloys.

3.7 *Results of porosity test*

Porosity test showed that whenever the hydrogen gas exceeded the 0.17 ml/100g value in the molten metal samples from casting launder, the pin holes were seen in the casting log samples directly with naked eye or under magnifying glass. It was also observed that porosity due to hydrogen was more prominent towards the periphery of the cast log sample. All the heats which contained H_2 less than 0.15 ml/100g at casting station showed no such problems and produced good quality castings. This test also confirmed the quantitative readings obtained from Leco RHEN-602 determinator.

4. CONCULSIONS

The absorption and accumulation of hydrogen gas in molten metal depends upon the raw materials, scrap %age and humidity level along with some other factors like natural gas purity etc. This study proved that Leco hydrogen determinator RHEN-602 can be used effectively for the detection of hydrogen contents in molten metal, and for the estimation of degassing efficiency. The average hydrogen removal efficiency of inline rotary degassing unit under study was lingered at 40%. Porosity test showed that H_2 values above 0.15 ml/100g in molten metal at casting station can cause serious damage to final castings.

5. ACKNOWLEDGEMENT

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