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Materials Science and Engineering A 428 (2006) 41–46

MATERIALS SCIENCE & ENGINEERING A

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On-line prediction of the melt hydrogen and casting porosity level in 319 aluminum alloy using thermal analysis

A. Mitrasinovic [a,*], F.C. Robles Hernández [b,c,1], M. Djurdjevic [d], J.H. Sokolowski [b]

[a] *University of Toronto, Department of Materials Science and Engineering, Walberg Bldg., Room 80, 184 College St., Toronto, Ont., Canada M5S 3E4*
[b] *NSERC/Ford-Nemak/University of Windsor Industrial Research Chair in Light Metals Casting Technology (IRC), Mechanical, Automotive and Materials Engineering, Essex Hall, 401 Sunset Avenue, Windsor, Ont., Canada N9B 3P4*
[c] *Transportation Technology Center Inc., TTCI Pueblo, CO 81001, USA*
[d] *Institute of Metallurgy, TU Clausthal, Clausthal-Zellerfeld, Germany*

Received 20 January 2005; received in revised form 12 April 2006; accepted 12 April 2006

Abstract

One of the major problems associated with aluminum castings is the porosity. Porosity is attributed to the shrinkage process and a lack of interdendritic feeding, which result in a reduction of the mechanical properties, loss of pressure tightness and poor surface integrity in castings.

In this research was developed a methodology for on-line prediction of the hydrogen and porosity levels in 319 melts and castings, respectively. The main tools used for this research were thermal analysis and image analysis. Thermal analysis revealed that the nucleation temperature for the Al–Si–Cu eutectic changed in up to 12.3 °C for a range of dissolved hydrogen from 0.06 to 25 mL H_2/100 g Al. The hydrogen–porosity threshold determined for the 319 alloy was ~0.15 mL H_2/100 g Al in the low pressure conditions (6 kPa). A statistical analysis demonstrated that this technology is highly accurate ($R^2 = 0.91$) to predict the amount of hydrogen and the porosity.

Keywords: Hydrogen level; Al–Si alloys; Porosity; Thermal analysis; Image analysis; Hydrogen threshold

1. Introduction

A comprehensive understanding of melt quality, including the porosity level, is very important for the control and prediction of casting characteristics. If one can assess these characteristics on-line during the manufacturing process, one can make proactive decisions pertaining to melt and casting quality control. This can substantially reduce cost downtime and scrap levels.

Hydrogen is the only gas soluble in liquid aluminum, and it is one of the main factors involved in the formation of porosity in aluminum castings. Quantification of the amount of dissolved hydrogen in aluminum melt has been for a long time a subject of high attention by several authors [1–10]. Among many developed techniques for the assessment of the amount of dissolved hydrogen into aluminum melt the Reduce Pressure Test (RPT) or Straube–Pfeiffer technique has been widely accepted at the aluminum foundry. The RPT utilizes a liquid aluminum sample that is poured into a pressure tight chamber. The sample is left there to solidify under low pressure. Under this condition, the growth of porosity is virtually unrestricted and the amount of porosity that is created by a given amount of hydrogen gas is much larger than one would expect under actual casting conditions. This makes it easy to qualitatively estimate the hydrogen content in the melt.

In the early stage of its development the RPT was only used as a quality control tool for molten alloy. Now conventional RPT is a semi-quantitative method for measuring hydrogen content and is widely used in the aluminum industry. It is simple, inexpensive and versatile method. Because it has various ways to show the existence and quantity of hydrogen, it has served a variety of purposes in production and research, such as quantity control and hydrogen detection and measurement. It has a workable accuracy for many applications. However, its accuracy is generally lower than that of some other methods, such as the sub-fusion and the fusion methods and Telegas.

The long time has been recognized a necessity of making the RPT truly quantitative measurement technique. In 1955 Rosen-

* Corresponding author. Tel.: +1 416 946 0421.
E-mail address: alex.mitrasinovic@utoronto.ca (A. Mitrasinovic).
[1] Formerly at the IRC, Canada and currently at TTCI, USA

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doi:10.1016/j.msea.2006.04.084

tal and Lipson [1] attempted to quantify the amount of dissolved hydrogen in aluminum melt by measuring the volume percent of porosity of the RPT samples. In experiments it was assumed that all of the pores formed in test sample under vacuum were filled with hydrogen gas at the applied pressure and solidification temperature. Sulinski and Lipson [5] have shown that assumption was not correct as some hydrogen was retained in solid solution while some was removed from the test samples during the test under a low pressure. During 1973 Hess [6] unsuccessfully tried to quantify the RPT by calibrating the test sample density against the amount of dissolved hydrogen.

Gruzleski and co-workers [7] showed that reduced pressure sample density could be related in a clear and reproducible way to the amount of hydrogen dissolved in aluminum melt and measured by one of the recirculation gas techniques. In that work Gruzleski and co-workers tried to develop a calibrated curve for a given alloy (413, 356, 357 and 319) and a specific sample mold geometry of the solidified test sample to the actual amount of dissolved hydrogen in liquid alloy. The following parameters have been analyzed in these experiments: sample pouring temperature, mold temperature, chamber temperature and pressure and melt cleanliness. The amounts of dissolved hydrogen were varied over the range 0.07–0.35 mL H_2/100 g Al melts by using various degassing and re-gassing procedures. In all cases a linear relationship was obtained to best fit the density–hydrogen and weight–hydrogen relationships. The correlation coefficient (R^2) for all experiments with various alloys falls in the range from 0.7 to 0.8. The scatter of the data according to authors can be related to the various parameters above mentioned. Among these variables the most important were variation in the melt cleanliness and imprecision in chamber pressure control. According to authors, the scattering of the data increases as the amount of dissolved hydrogen increases. For the entire hydrogen range used in their experiments the amount of 0.15 mL H_2/100 g Al melt has been recognized as a threshold value. This value represents the amount of dissolved hydrogen below which it is not reasonable to detect any gas pores in as cast solidified structure using conventional metallographic techniques by visual observation.

Cooling curve analysis has been used for many years to define binary phase diagrams and for fundamental metallurgical studies [8–12]. The cooling curve method is used in commercial applications for characterization of the melt, for instance, the level of silicon modification, the low melting points of secondary eutectic(s), fraction solid and other characteristic temperatures such as liquidus, Al–Si eutectic, Al–Si–Cu eutectic and solidus temperature. Thermal analysis was also used to determine and predict casting properties including grain size, dendrite arm spacing (DAS) and dendrite coherency point. In addition thermal analysis is a simple, inexpensive and a consistent technique with high level of accuracy and repeatability.

Recently, it has been proved that thermal analysis (TA) can be used to assess the level of dissolved hydrogen in aluminum melt [13]. It was found that an increase in the hydrogen levels from 0.083 to 0.280 mL H_2/100 g Al decreases the $T_{E,NUC}^{AlSiCu}$ temperature of the 319 aluminum alloys by 8 °C. Statistically high correlation coefficient ($R^2 = 0.82$) between $T_{E,NUC}^{AlSiCu}$ and the hydrogen dissolved in the aluminum melt suggests that dissolved hydrogen depresses the nucleation temperature of the copper rich eutectic phases. This difference is large enough to be used as a parameter for predicting the level of dissolved hydrogen in the liquid aluminum melt. The result is expected since the literature suggests that copper decreases the hydrogen activity in liquid aluminum alloys [4,5]. Therefore, it is plausible to note that an increase in the concentration of hydrogen increases copper's ability to react with Al, Si and Mg which results in the formation of copper rich eutectic phases at lower temperatures.

Unfortunately, there are no data in the literature showing that thermal analysis can predict the amount of porosity in cast aluminum alloys. Therefore, the main purpose of this work is to find out if thermal analysis in combination with RPT technique can be used for on-line prediction of the amount of porosity in aluminum cast components. The present work was partly inspired by the earlier attempts of the authors to develop a quantitative technique for assessment of the amount of porosity and dissolved amount of hydrogen in 3XX series of aluminum alloys using RPT apparatus.

2. Experimental procedures

2.1. Materials

Secondary W319 aluminum ingots from the Nemak-Windsor Aluminum Plant production line were used in all experiments. The chemical composition as obtained from the optical emission spectroscopy for the alloy used in the present research is provided in Table 1.

2.2. Melting and degassing procedures

The secondary W319 aluminum ingots were cut and loads of 12 kg were charged into the crucible of a resistance furnace. In order to eliminate the moisture, the ingots were preheated in the resistance furnace at 400 °C for few hours. Finally, the alloy was heated up to 760 °C. The melt temperature was chosen to be constant parameter during all experiments. The temperature of the melt was controlled with a thermocouple inserted into the liquid metal, while the chamber temperature of the furnace was controlled with the thermocouple attached to the inner wall of the furnace. The top of the furnace was covered with high temperature resistant bricks in order to eliminate any temperature gradient in the molten alloy.

A degassing unit (FOSECO) was used to reduce the level of hydrogen in the melt on the lowest feasible level. Following the removal of the dross, the graphite propeller was introduced into the melt and the top of the furnace was again protected. Propeller rotated 100 cycles/min blowing argon at a rate of

Table 1
Chemical composition (wt%) for the W319 alloy used in the Enviro-AlTAP experiments

Si	Cu	Fe	Mg	Mn	Zn	Ti	Ni	Sn	Pb
7.77	3.48	0.42	0.17	0.25	0.18	0.12	0.04	0.04	0.008

0.28–0.34 m^3/h. The degassing process took 20 min, at which time the hydrogen level reached its lowest level.

2.3. Hydrogen measurement

Hydrogen measurement was made using calibrated AlSCAN unit. The top of the furnace was protected and covered in order to reduce the loss of heat and eliminate additional dissolution of hydrogen into the aluminum melt. The temperature during hydrogen measurements was kept constant at 760 ± 5 °C. Each hydrogen measurement was repeated three times. If the similar values for the amount of dissolved hydrogen have been obtained in all readings, the last one was taken for further consideration (analysis). In the case of different values, the readings have been repeated as many as necessary times in order to satisfy criterion of repeatability.

2.4. Thermal analysis procedure

Thermal analysis experiments were conducted using the Enviro-Aluminum Thermal Analysis Platform (Enviro-AlTAP) developed at the University of Windsor. After the hydrogen content was recorded, the W319 test samples with masses of 300 ± 5 g were poured into a stainless steel test cup with a preheated ladle (Fig. 1). The bell, with the previously prepared and connected K type thermocouple, was placed over the cup containing the liquid metal, and thermal analysis test samples were solidified at different vacuum levels. The level of vacuum was maintained at 0 and 673 mmHg (101 and 6 kPa). The triggering temperature for the beginning of the thermal analysis was 730 °C. National Instruments data acquisition system linked to a personal computer having the software capacity of analyzing up to 50 cooling curves and its derivative parameters has been used in these experiments.

Previous experiments by the IRC team have shown that certain parameter(s) from the cooling curve(s) can be used to assess the amount of dissolved hydrogen in the liquid aluminum melt. This paper tries to analyze how do different pressures (from 6 to 101 kPa) and amount of dissolved hydrogen (from 0.06 to 0.25 mL H_2/100 g Al alloy) influence the cooling curve characteristics and which parameter(s) from the cooling curve can be used to assess the amount of porosity in as cast aluminum structure.

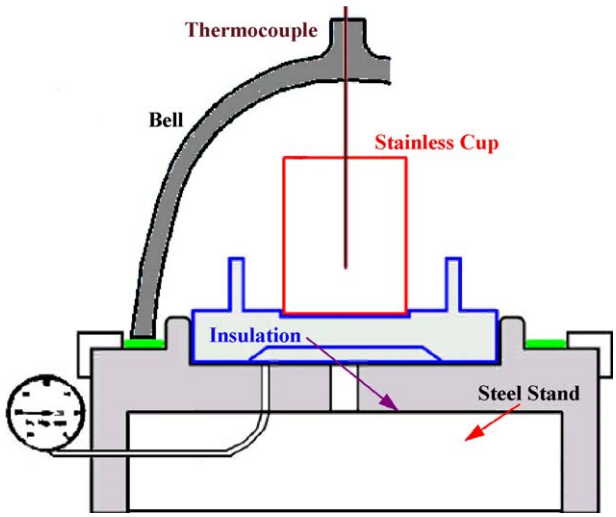

Fig. 1. Enviro-AlTAP platform.

Two levels of hydrogen were studied in this paper: the low level which is below 0.15 mL H_2/100 g Al and the high level which is above 0.15 mL H_2/100 g Al. The applied pressure was also investigated at two levels. Experiments performed at an atmospheric pressure of 101 kPa were considered to be low vacuum level conditions. Experiments performed at 6 kPa were considered as the higher vacuum level.

A total of 32 (16 under low pressure and 16 under atmospheric pressure) laboratory thermal analysis test samples were collected

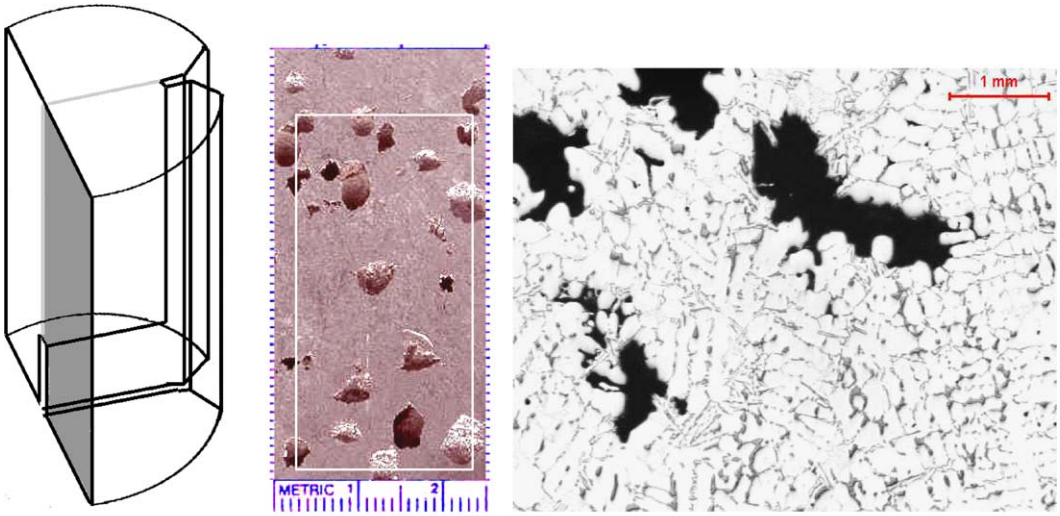

Fig. 2. Area percent porosity analysis: (a) schematic drawing of the sample showing the preliminary preparation for metallography analysis; (b) analyzed surface; (c) microstructure showing a field taken using image analysis.

for further metallographic sample preparation and image analysis.

2.5. Metallographic analysis

Enviro-AlTAP test samples were prepared for metallographic analysis and the area percent of porosity was measured. Samples were cut longitudinal and the dark grey surface in Fig. 2a was prepared for metallographic observation. Forty-eight fields at a magnification of 16×, covering an area of 40 mm × 20 mm (Fig. 2b), were measured in every test sample. Electronic micrographs were taken using the Leica Optical microscope. Area percent porosity was marked manually in every figure acquired from the microscope (Fig. 2c). Image analysis system made automatic calculation of area percent porosity for every field. Total of 48 fields with area percent porosity measurements for every sample were recorded in a computer file. The average value of 48 fields was accepted as the area percent porosity for a given thermal analysis test sample.

3. Results and discussion

The quality of aluminum alloys is determined by their microstructures and the amount of porosity present in the as cast structures. Porosity is a leading cause in the reduction of their mechanical properties particularly fatigue resistance. In recent years great emphasis has been located on quality control of cast aluminum alloys. Hydrogen content in liquid aluminum alloys is one of the factors which must be controlled since the level of porosity in solidified cast aluminum products strongly depends on the amount of hydrogen soluble in liquid aluminum. Previous IRC research [13] verified that some cooling curve characteristics could be used for quantitative analysis of the hydrogen dissolved in the aluminum melt. This was the first scientific analysis for hydrogen in aluminum alloys using the thermal analysis technique. The results of this work encourage authors to believe that the integrated thermal analysis technique(s) can be developed to address the complex quality control requirements for most metal casting operations. Therefore, statistically designed experiments were performed by the IRC at the University of Windsor with different amounts of dissolved hydrogen (0.063–0.225 mL H_2/100 g Al melt) to find out the parameter(s) that can be used to assess the level of porosity in as cast aluminum structure. More than 40 parameters from the cooling curve were statistically analyzed in order to find the most significant one that could be used as a criterion for the prediction of dissolved hydrogen in the W319 aluminum melt. Among them three parameters were related to characteristic nucleation temperatures, which are the liquidus or α-Al nucleation, the Al–Si eutectic and the Al–Si–Cu eutectic temperatures ($T^{\alpha DEN}_{NUC}$, $T^{AlSi}_{E,NUC}$ and $T^{AlSiCu}_{E,NUC}$, respectively). These parameters showed statistical significance related to different amounts of dissolved hydrogen in the aluminum melt.

Experimental and statistical analyses reveal that liquidus temperatures ($T^{\alpha DEN}_{NUC}$) decreased slightly with increasing hydrogen level. The same tendency was observed for the Al–Si eutectic nucleation temperature ($T^{AlSi}_{E,NUC}$). The difference in $T^{AlSi}_{E,NUC}$ between tests with high and low hydrogen levels was 5.52 °C. This difference is greater than that for liquidus temperatures but still not large enough for the technique to be used at the plant level for the determination of hydrogen level in W319 aluminum melt. The increases of hydrogen from 0.06 to 0.22 mL H_2/100 g Al melt resulted in a reduction of the $T^{\alpha DEN}_{NUC}$ and $T^{AlSi}_{E,NUC}$ by 2.89 and 5.52 °C, respectively.

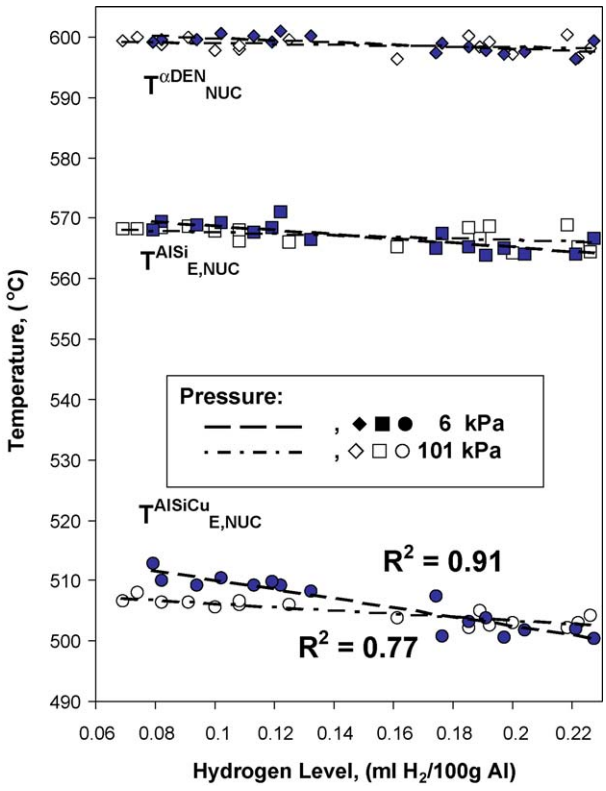

Fig. 3. Relationship between characteristic temperatures of solidification of the W319 aluminum melt and amount of dissolved hydrogen.

However, the effect of different amounts of hydrogen on the $T^{AlSiCu}_{E,NUC}$ temperature is much more significant. As can be seen in Figs. 3 and 4, under normal atmospheric pressure, the addition of 0.18 mL H_2/100 g Al melt aluminum decreases the $T^{AlSiCu}_{E,NUC}$ by approximately 6 °C. This effect is even more significant when the thermal analysis sample was solidified at low pressure (6 kPa). In this case the reduction of the $T^{AlSiCu}_{E,NUC}$ for the same addition of hydrogen was 12.3 °C. In addition, the highest slope and the best-fit line corresponded to the results obtained using Enviro-AlTAP at low pressure conditions. This means that the nucleation reaction with the highest change, thus sensibility with the amount of dissolved hydrogen into liquid aluminum melt, can be used to precisely quantify the hydrogen. Table 2 shows that experimental data collected from the thermal analysis (cooling curve) of the sample solidified at low pressure. The statistical analysis demonstrated that experiments conducted using Enviro-AlTAP at low pressure conditions resulted more significant ($R^2 = 0.91$) in comparison to those that were collected during solidification under atmospheric (101 kPa) pressure ($R^2 = 0.77$). In fact, this was expected since the literature suggests that copper decreases the hydrogen activity in liquid aluminum alloys [8,9].

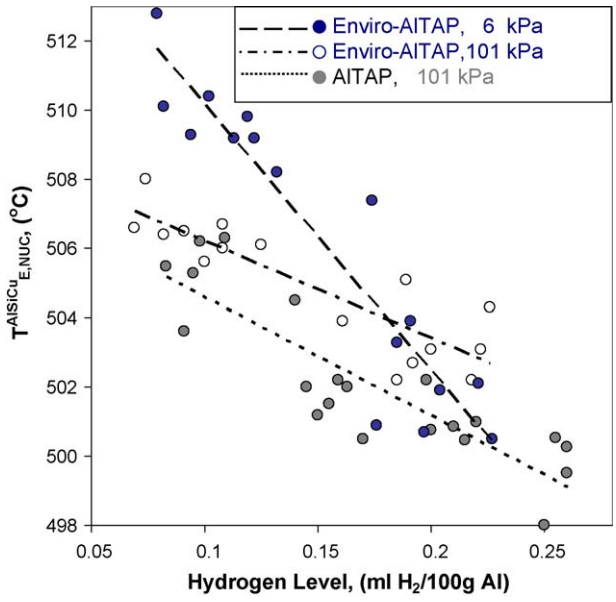

Fig. 4. Assessment of the hydrogen level(s) using the AlTAP and Enviro-AlTAP platforms.

Therefore, it is plausible to note that an increase in the concentration of hydrogen increases copper's ability to react with Al, Si and Mg which results in the formation of copper rich eutectic phases at lower temperatures.

Previous analysis conducted by Crossley et. al [9] was in good correlation to the results obtained by the IRC using the Enviro-AlTAP technique at atmospheric pressure. However, in this research investigation the correlation coefficient (R^2) for measured $T^{AlSiCu}_{E,NUC}$ was considerably higher because the samples were solidified at lower cooling rate, using thinner test cup and the surrounding atmosphere was carefully controlled.

The visual observation of a cut specimen solidified under vacuum is widely used at the foundry floor for qualitative control of as cast products because it is simple and fast. Density measurement of a test samples solidified under low pressure is an often used method for qualitative assessment of the level of porosity in cast components. Unfortunately, the density measurement is not enough accurate, cannot produce the reliable results and cannot be used for on-line quality control. The difference in the density measurement of samples solidified under reduce pressure was more than 25% between two measurements conducted within 24 h [14]. It was also found that the divergence of the data was greater at higher hydrogen contents than at the lower ones. The higher divergence for the experiments with higher hydrogen could be due to the fact that the vacuum removed some of the hydrogen.

Table 2
Multiple correlation coefficients (R^2) for measured $T^{AlSiCu}_{E,NUC}$ by different systems for assessment of the hydrogen level in the W319 aluminum melt

System	Level of hydrogen (mL H_2/100 g Al)	R^2
AlTAP	$-0.0222 \times T^{AlSiCu}_{E,NUC} + 11.327$	0.76
Enviro-AlTAP (101 kPa)	$-0.0274 \times T^{AlSiCu}_{E,NUC} + 13.961$	0.77
Enviro-AlTAP (6 kPa)	$-0.0114 \times T^{AlSiCu}_{E,NUC} + 5.8994$	0.91

Table 3
Average area percent porosity of Enviro-AlTAP samples

Pressure	Low Hydrogen (0.060-0.125 ml H_2/100g Al)		High Hydrogen (0.126-0.225 ml H_2/100g Al)	
		Area % Porosity		Area % Porosity
101 kPa		0.94		0.97
6 kPa		3.79		10.87

Among other available methods, the image analysis is the one that provides an accurate porosity level from the analytical surface. Table 3 shows the average porosity content as measured by image analysis for the different hydrogen levels and applied pressures. Fig. 5 shows the considerable increase in the porosity content when the amount of dissolved hydrogen in aluminum melt increases beyond 0.15 mL H_2/100 g Al melt.

Fig. 5 shows that thermal analysis and image analysis results can be combined together and used to assess the level of porosity in solidified as cast aluminum components. As previously mentioned, $T^{AlSiCu}_{E,NUC}$ shows the highest sensitivity to the hydrogen level in the 3XX aluminum melt. Fig. 5 illustrates that the area percent porosity and the $T^{AlSiCu}_{E,NUC}$ have the same tendency. This observation is consistent with the literature data, where it is known that for any given alloy and specific solidification conditions there is a "threshold hydrogen content" below which no observable gas based primary porosity is formed [15,16]. The threshold of hydrogen dissolved in aluminum melts for thermal analysis samples that solidified under atmospheric pressure was 0.18 mL H_2/100 g Al melt (see Fig. 5).

As can be seen in Fig. 5 the area percent of porosity for experiments conducted under atmospheric pressure increases linearly with respect to hydrogen content. This increase has different paths for the experiments conducted under low pressure conditions with very distinguished change in the slope which corresponds to 0.14 mL H_2/100 g melt.

Ransley and Neufeld [17] found that porosity did not form below hydrogen content of 0.12 mL H_2/100 g Al. They described the fact that the hydrogen content for 0% porosity was observed to be three times higher than solid solubility limit, 0.04 mL H_2/100 g Al, to the existence of a required degree of super saturation before the formation of the gas pores would take place. Deoras and Kondic [18] noted that the threshold of dissolved hydrogen in aluminum alloys was 0.15 mL H_2/100 g Al; in addition, they did not found or reported porosity for the Al–6% Si alloy at hydrogen levels of up to 0.13 mL H_2/100 g Al.

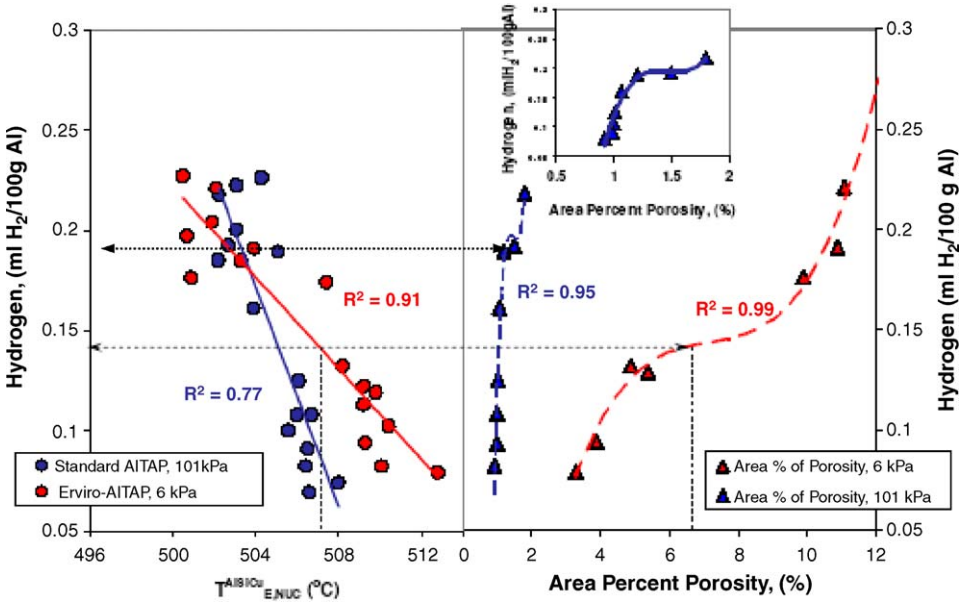

Fig. 5. Correlation between $T^{AlSiCu}_{E,NUC}$ and area percent porosity as a function of the hydrogen level and applied pressure.

Anson and Gruzleski [15,16] explain that during formation of microporosity, nucleation and growth occur continuously over a wide time/temperature range. Little porosity exists until 0.17 mL H_2/100 g Al, after which the percent porosity increases linearly with the hydrogen content (the threshold hydrogen content), for a threshold of hydrogen lower than the 0.1 mL H_2/100 g Al has been usually accepted and considered to form none or negligible porosity.

Before and after the threshold of hydrogen, the pore density behaves differently showing a dramatic increase for hydrogen levels above the threshold. Since there is a large change in pore density at the threshold, the nucleation of the pores is shown to be partly dependent on the hydrogen content. Work by Tynelius [19] has shown the number of pores is dependent on the solidification rate. In Whittenberger and Rhines [20] work from 1952, a vacuum induction furnace was used to melt the alloy and the molten metal was used to cast samples that were solidified in vacuum. They showed that if the melt was free of hydrogen then the ingot would be altogether free from porosity.

4. Conclusions

Thermal analysis and the Enviro-AlTAP technologies demonstrated to be reliable technologies to measure the level of dissolved hydrogen for Al–Si alloys. Using the above-mentioned technologies was found that three nucleation reactions ($T^{\alpha DEN}_{NUC}$, $T^{AlSi}_{E,NUC}$ and $T^{AlSiCu}_{E,NUC}$) were affected by the amount of dissolved hydrogen during the solidification process. The change in the nucleation temperatures for the previously mentioned reactions was 2.89, 5.52 and 12.3 °C, respectively, in comparison to low and high levels of dissolved hydrogen (for 0.06 to 25 mL H_2/100 g Al). Therefore, it was considered that the temperature of nucleation of the Al–Si–Cu ($T^{AlSiCu}_{E,NUC}$) reaction presented the highest response with the amount of dissolved hydrogen. Furthermore, using low vacuum in the Enviro-AlTAP apparatus the correlation coefficient was further improved from $R^2 = 0.77$ to 0.91. In addition, the characteristic threshold of dissolved hydrogen for the W319 Al–Si alloy was ~0.14 mL H_2/100 g Al.

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