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Comparison of microstructure and mechanical properties of industrial pure aluminum produced by powder metallurgy and conventional rolling

Comparación de microestructura y propiedades mecánicas de aluminio industrialmente puro fabricado por laminado convencional y metalurgia de polvos

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Abstract

Aluminum alloys are produced by different manufacturing techniques and each technique produces a unique microstructure. One of the most versatile manufacturing methods is powder metallurgy (P/M) because it produces a final fine or ultrafine grain microstructure. But it requires powder consolidation and densification processes after P/M. In the other side, Hot Isostatic Pressing (HIP) is now recognized as a very interesting technique to densify advanced materials with a high potential. The aims of this work are: to produce an AA1100 aluminum billet by powder densification followed by HIP and to compare its microstructure and mechanical properties against the ones of an AA1100 aluminum plate produced by conventional rolling. The mechanical properties were measured using tensile test and Charpy test. The results show that the tensile strength of the P/M+HIP sample is higher than the one of the aluminum plate, but the yield strengths are similar. In the other hand the impact toughness results are very different from each other.

Alloy, Microstructure, Property

Resumen

Las aleaciones de aluminio se producen mediante diferentes técnicas de fabricación y cada técnica produce una microestructura única. Uno de los métodos de fabricación más versátiles es la metalurgia de polvos (P/M) porque produce una microestructura final de grano fino o ultrafino. Sin embargo, este método requiere de procesos de consolidación y densificación de polvo posteriores. El prensado isostático en caliente (HIP) es una técnica ampliamente utilizada para densificar materiales obtenidos por P/M, fundiciones comerciales y los nuevos materiales producidos por manufactura aditiva. Los objetivos de este trabajo son producir un tocho de aluminio AA1100 por densificación de polvo seguido del proceso HIP para comparar su microestructura y las propiedades mecánicas contra las de una placa de aluminio producida por método convencional. Las propiedades mecánicas fueron medidas mediante ensayo de tracción y ensayo Charpy. Los resultados muestran que la resistencia a la tracción de las muestras de P/M+HIP es mayor que las muestras de la placa de aluminio, pero los límites elásticos son similares. Por otro lado, los resultados de la tenacidad al impacto difieren fuertemente entre ambos materiales.

Aleación, Microestructura, Propiedades

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Introduction

Aluminum alloys are in great demand in various industrial sectors, as they offer a good agreement between strength and density, as well as being relatively cheap [1]. Aluminum alloys are large family of alloys with a variety of alloying elements, but not all Al alloys behave similarly when they are processed by the different manufacturing methods due to the difference in alloy elements and compositions [1-3]. There are many processing routes to produce aluminum alloys and each of them leads to a unique microstructure and mechanical properties. Among all the manufacturing techniques one of the most versatile is powder metallurgy (P/M), because it allows to alloy beyond the equilibrium concentration different metals and no metals, while at the same time it produces a final microstructure with fine or ultrafine grain structures. It P/M has been successfully applied for alloying aluminum with elements such as: Si, Al, Cu, Mg, etc. but also to fabricate nanocomposites of aluminum with different reinforcement particles such as yttrium oxides, aluminum oxides, carbon nanotubes, etc. [4-12].

The high energy grinding process produces the formation of agglomerates and by controlling the processing parameters it is possible to reduce the amount and size of said agglomerates, which translates into an increase in the production efficiency of processed powders. The processing of powders through cycles at high and low revolutions favors control in agglomerates as a result of the interactions between balls and particles [13-17].

Powder metallurgy requires powder consolidation and densification processes after the powder particles are processed in a high-energy ball mill. Some of the densification techniques applied for getting useful products are: strained powder rolling, sinter forging, or Hot Isostatic Pressing (HIP). Among those processes, HIP is now recognized as a very interesting technique to densify advanced materials with a high potential and it is the way to optimize material properties [7- 10].

HIP is defined in ASTM B998-17 as a process were components are subjected to the simultaneous application of heat and high pressure in an inert gas medium [11].

The process is used for the reduction of internal (non-surface connected) porosity. It is considered as efficient method for improving mechanical properties for a great variety of alloys and it is recommended also for additive manufacturing components. In this way, HIP is becoming the standard method to improve lifetime of critical parts. Common applications for HIP in the industry are: closed porosity removal, consolidation of powders and diffusion bonding of dissimilar metals or alloys [7-13].

In literature there is much information about composites, nanocomposites and aluminum alloys powders consolidated using HIP [4-12]. However, there is too few information about consolidated pure aluminum powders using this method and its final mechanical properties. Therefore, the aims of this work are: to produce an AA1100 aluminum billet by powder consolidation in Hot Isostatic Pressing (HIP) and to compare the microstructure and mechanical properties measured using tensile test and Charpy test against the ones of aluminum AA1100 plate produced by conventional rolling.

Methodology

In this work, the aluminum powders were processed in a high-energy ball milling (HEBM) commercially named as Simoloyer CM01 with capacity of 2000 ml in volume (figure 1). The grinding parameters used were 850rpm/5min and 250rpm/1min until reaching 50 minutes of processing. In this case, only the time at high speed was considered as process time. Figure 1 shows the HEBM system with the adaptations for the control of the atmosphere prior to start of the process, as well as the control program.

The processed powders were passivated in a specially designed and manufactured for this purpose container with a controlled amount of oxygen. The operation was carried out in vacuum within the container that was located on rotating rollers. Then air was slowly introduced in several stages. Since the passivation process is exothermic, the temperature in the system need to be monitored. This process was finished when no change in the temperature was registered. The rotation by means of the rollers allowed the homogeneous passivation of the powders.

Powders processed by HEBM have different morphology as raw materials and particle size distribution is normally broad. Therefore it is necessary operations as sieving in order to get the suitable distribution particles sizes for sintering using HIP. For the sintering of aluminum powders, particles larger than 250 microns in size were separated, because they make consolidation difficult due to the increase in spaces among particles, which leads to greater interaction distances among them and generating porosity in the solid component.

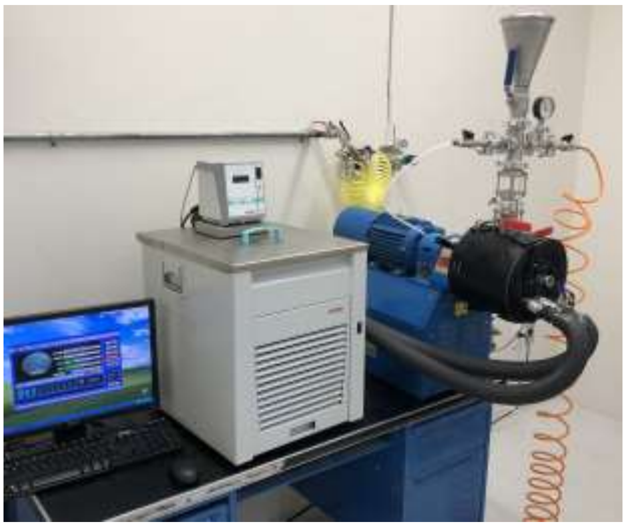


Figure 1 HEBM e equipment for powders processing

Powder encapsulation required a series of sequential operations that determine the success of powder consolidation by means of HIP process. The procedure activities are depicted in figure 2 and 4:

Figure 2 Welding of the powders containers for sealing the container

Figure 3 PT to verify the integrity of the container´s welding

After verifying that the encapsulation did not have porosity throughout the weld and it maintained a vacuum, consolidation was carried out by HIP.

Dye Penetrant testing (PT) and vacuum testing were carried out for assessing if the powder encapsulation was sealed.

The consolidation of the powders by HIP had as process parameters: temperature, time and isostatic pressure of the argon gas used. In this work a Quintus Hot Isostatic pressing equipment was employed for consolidation system and the parameters were: 550 °C, 125 MPa and 5 hours. The results obtained from the HIP treatment of the encapsulated powders with the selected treatment parameters are shown in Figure 4.

Figure 4 Billet produced by HIP densification and transversal cut.

The visual inspection of the container indicated deformation, which meant a successful sintering of powders.

The integrity of the weld throughout the package was also assessed by visual testing. The upper and lower covers of the encapsulation were removed by mechanical cuts. From this point, this billet is denominated as P/M+HIP Billet. The P/M+HIP billet obtained was nondestructive inspected using an X-ray microfocus CT cabinet system for 3D metrology and analysis model PHOENIX V/GTOME/XM.

For comparison an AA11010 aluminum plate was used. The chemical composition was determined by the same techniques applied in the P/M+HIP billet. The chemical composition of the aluminum plate, and the powder encapsulation was determined by Optical emission spectrometry using an OBLF Optical emission Spectrometer QSN-750-II.

The mechanical properties were measured using a universal testing machine SHIMADZU WADE AGX-V 100KN following ASTM E8/E8M-21. For this tests a class B extensometer was used. The Charpy test was carried out using a SATEC model CHARPY impact pendulum according to ASTM E23/E23-18. Samples for tensile and Charpy impact testing were taken at 0°, 45 ° and 90° from the rolling direction of the AA110 aluminum plate; but due to the dimensions of the aluminum P/M +HIP billet only sub size samples were taken.

Samples were cut from the aluminum plate and the P/M+HIP billet for metallographic preparation following ASTM E37E3M -17. After that they were mounted in conductive Bakelite and grinded from 120 to 2000 SiC grinding paper. The samples were polished using 1 µm alumina powder slurry and a final polishing was carried out using diamond paste as polisher. A final etching step was performed using Keller´s as a reagent for revealing the microstructure. The observation was made with a NIKON EPIPHOT 200 optical microscope was carried out using a Nikon Optical microscope with ZEISS image analysis. Fractographic analysis of the Charpy and tensile tested samples was carried out using JEOL JSM 6610LV scanning electron microscope (SEM).

Results

The chemical composition of the aluminum AA1100 plate produced by conventional rolling and the P/M+HIP billet were determined. The results are shown in table 1, there is seen that the content of alloying elements shows small differences but the aluminum weight percentage was the same in both cases.

	% Cu	% Fe	% Mn	% Si	%Zn	% Ti	% Al
AA1010 standard	0.05-0.20	0.05 max.	0.05 max	NA	NA	NA	99.80 - 99.655
Al billet	0.00	0.01	0.00	0.11	0.02	0.01	99.71
AA1100 plate	0.00%	0.16	0.01	0.07	0.01	0.01	99.71

Table 1 Chemical composition of the aluminum AA1100 plate produced by conventional rolling and a billet produced by HIP

The P/M+HIP billet was nondestructive inspected by an X-ray microfocus CT cabinet system and the results showed evidence of aligned porosity of both types: connected and no connected (figures 5 to 7). Moreover, neither cracks nor laminations were detected. The results are shown in figures 6 and 7.

The microstructure of the P/M+HIP billet is seen in figure 8. There are seen fine grain size equiaxied grains and no evidence of precipitates nor second phases. There is seen good densification of the powders. Besides no evidence of connected porosity was found in the microstructure.

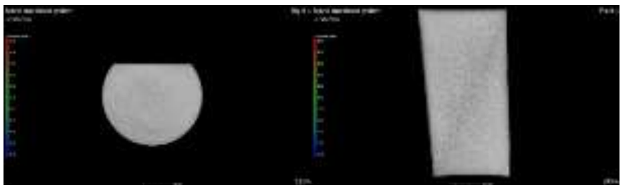


Figure 5 Images of the billet produced by HIP using X-ray microfocus CT cabinet system for 3D metrology and analysis model PHOENIX V/GTOME/XM

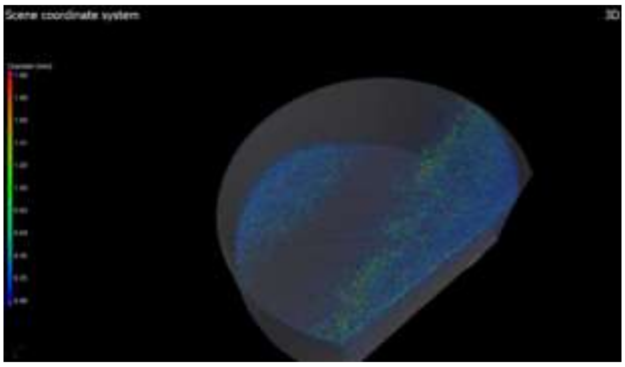


Figure 6 Image of the billet produced by HIP using X-ray microfocus CT cabinet system for 3D metrology and analysis model PHOENIX V/GTOME/XM

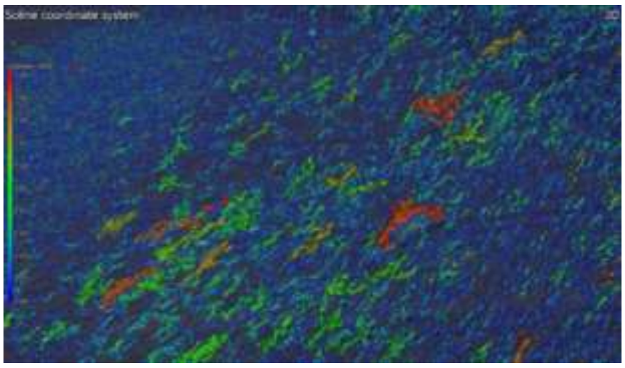


Figure 7 Image of the billet produced by HIP using X-ray microfocus CT cabinet system for 3D metrology and analysis model PHOENIX V/GTOME/XM.

Figure 9 shows the microstructure of the AA1100 aluminum plate produced by conventional method. There are seen equiaxial grains and no second phases. This microstructure matches with the reported information in literature. Room temperature tensile tests were conducted to determine mechanical properties following ASTM E8/E8M-21. Moreover, the impact toughness was measured by using the standard for the Charpy V-notched bar impact tests following E23/E23-18. The results of tensile test are shown in graphic 1 and table 2.

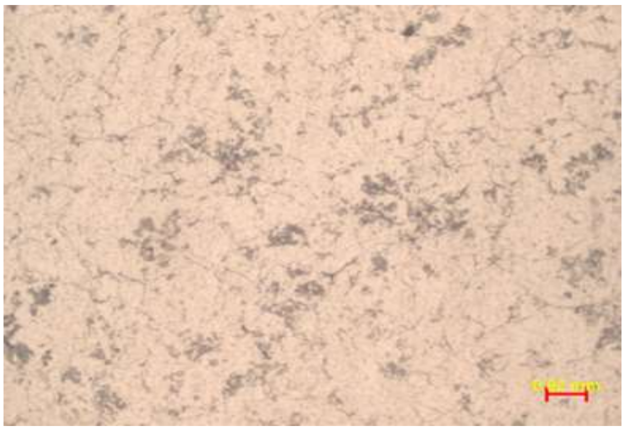


Figure 8 Microstructure at 500x of the billet produced by P/M+HIP

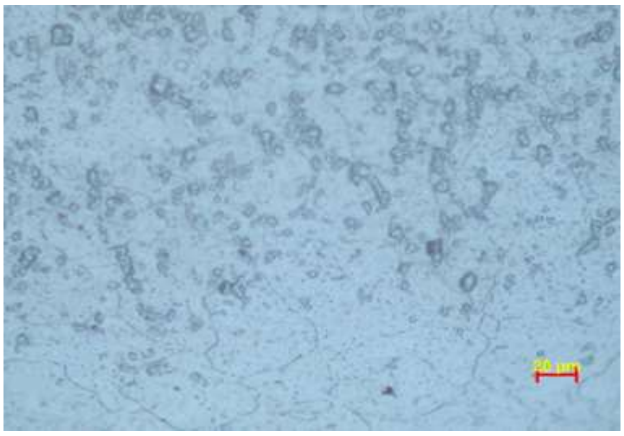
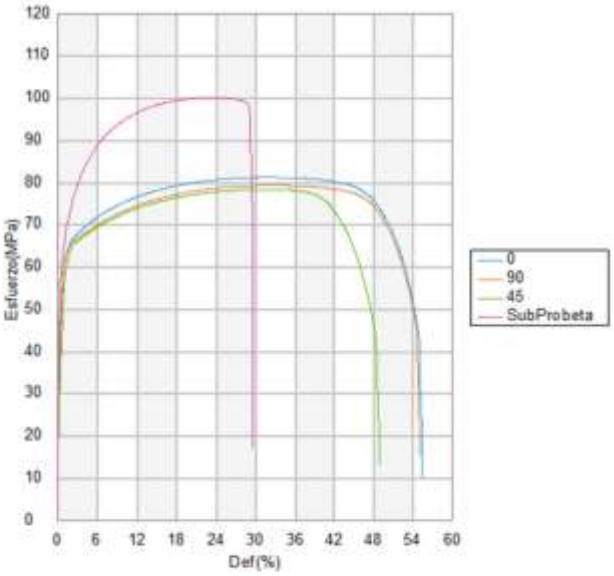


Figure 9 Microstructure at 500x of the AA1100 aluminum plate produced by conventional rolling



Graphic 1 Stress-strain curves of the aluminum plate(0°, 45 ° and 90° from the rolling direction) and the PM+HIP samples

Table 2 shows the average mechanical properties of the samples. There is seen that no big differences existed among the samples taken from the aluminum plate but there is a big differences between the mechanical properties taken from samples of the plate and the P/M+HIP samples.

	UTS (MPa)	Yield (MPa)	Lo (mm)	Lf (mm)	% Elong.
0	81.1135667	61.8447	50.00	81.16	62.32
45	78.0940333	61.4595	50.00	81.715	63.43
90	78.9505333	60.5781	50.00	81.0766667	62.15
P/M+HIP	100.024	59.3995	25.00	32.43	29.72

Table 2 Results of the mechanical properties measured using tensile test

Table 3 shows the impact toughness measured using Charpy impact test. There is a big difference between the between the absorbed energy of samples taken from the plate and the samples taken from the P/M+HIP.

Sample	Joules (average)
Plate 0° rolling direction	70.5
Plate 45° rolling direction	65.5
Plate 90° rolling direction	65.5
P/M+HIP	0.7

Table 3 Results of the impact toughness measured using Charpy impact test

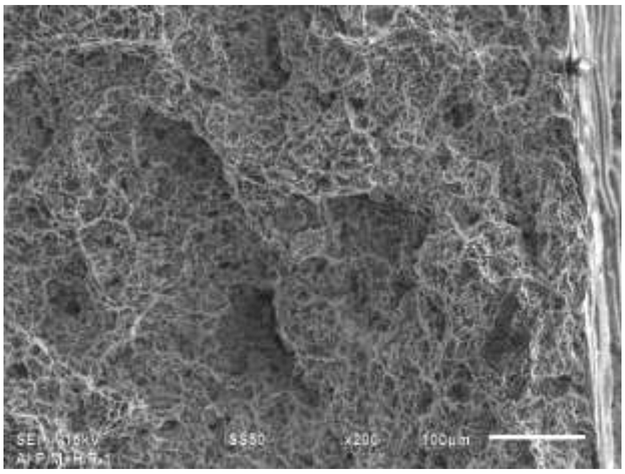


Figure 10 The SEM image shows the fracture Surface of the PM+HIP sample after Charpy testing

Figure 10 shows the fracture surface of the PM+HIP sample after tension testing. There is seen dimples, pores, and inclusions. Figure 11 shows the fracture surface of the PM+HIP sample after Charpy testing. There is seen dimples, pores, and inclusions.

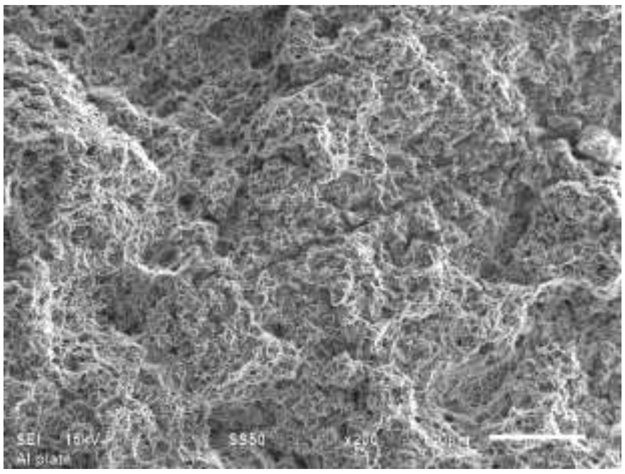


Figure 11 The SEM image shows the fracture Surface of the PM+HIP sample after tensile testing

Figure 12 shows the fracture surface of the plate with conventional rolling sample after Charpy testing.There is seen shearing dimples. Figure 13. The SEM image shows the fracture Surface of the plate with conventional rolling sample after tensile testing. There is seen tensile dimples.

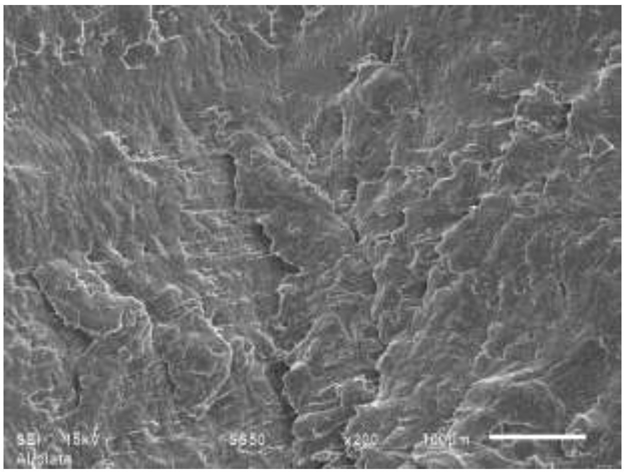


Figure 12 The SEM image shows the fracture Surface of the plate with conventional rolling sample after Charpy testing

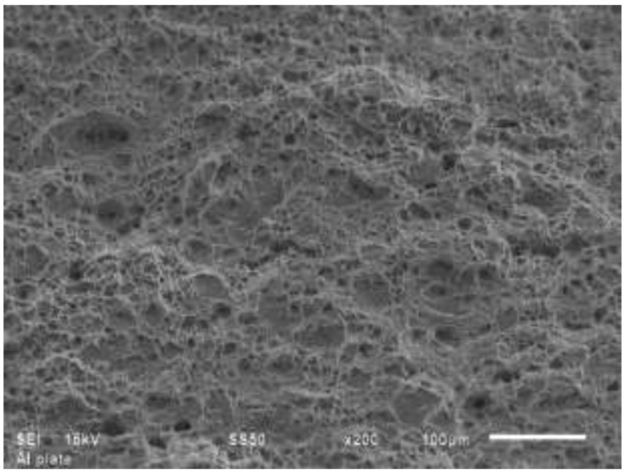


Figure 13 The SEM image shows the fracture Surface of the plate with conventional rolling sample after tensile testing

Discussion of results

The results of the chemical composition indicated that there is no big difference between the samples taken from the plate and the samples taken from the P/M+HIP. Both materials can be denominated as AA1100 aluminum. However, it must be kept in mind that the 1100 aluminum is almost pure aluminum but it is prone to strain hardening if it contains appreciable amounts of impurities such as iron and silicon. In the other hand, when this material is heated, the grain size might grow and the final grain size is influenced by impurities such as Cu, Fe, Mg, and Mn. In both cases there is an influence of the impurities in the microstructure and on the final mechanical properties of the material [1-3]. In this case the very low amount of impurities is an indication that no real influence of the impurities might be expected in the final mechanical properties of the aluminum billet P/M+ HIP.

Nondestructive evaluation (NDE) techniques are utilized to look for internal porosity and establish control samples with known porosity size and distribution. In this case, the nondestructive inspection using X-ray microfocus CT cabinet system for 3D metrology and analysis model PHOENIX V/GTOME/XM was applied to the P/M+HIP aluminum billet. The results showed evidence of aligned and connected porosity in the interior of the P/M+HIP aluminum billet. This fact was important because the samples for mechanical testing were cut in different position of the porosity. Furthermore, it was interesting to find that the porosity was located near the external surface and in specific areas. This is clearly seen in figure 7. This fact matched with the information reported in literature, which stated that the skill of a HIP cycle to completely close all porosity in a material is dependent on the severity of the porosity and on the HIP parameters [12-14, 22, 23]. However, the evidences of the nondestructive inspection are indications of the effectiveness of HIP and the mechanical integrity. The last one is strongly influenced by the location and type of defects.

Figure 9 shows the typical microstructure of the AA1100 aluminum cold rolling plate, which consists of equiaxial grains of aluminum. There was observed no evidence of segregates. In the other hand, figure 8 shows the microstructure of the P/M+HIP billet, there is seen fine equiaxial grains with no evidences of porosity nor precipitates. However, the grain size of the aluminum plate is bigger than the one of the P/M+HIP billet. The differences in microstructures exhibited in the two aluminum samples processed by two different methods indicated a difference in mechanical properties. This statement was probed by the tensile tests and impact Charpy test.

The tensile mechanical properties showed that the yield strength of the aluminum plate and the P/M+HIP billet sample were very similar, around 60 MPa, which can be observed in table 1. It suggested that HIP has little effect on the yield strength. However, there was a relevant difference concerning the ultimate tensile strength (UTS) of both materials. The aluminum plate was 80 MPa in UTS, which is the reported tensile strength of this type of aluminum with no internal defects. Meanwhile, the P/M+HIP billet was around 100 MPa.

This difference is explained by the difference in grain sizes; if one keeps in mind the Hall-Petch relation between grain size and mechanical properties.

Contrary to the tensile mechanical properties, the impact toughness, measured using Charpy impact test, of the P/M+HIP billet was much lower than the one of the aluminum plate. This results confirm a brittle behavior of the P/M+HIP aluminum, which is opposite to the behavior of the aluminum plate. It is well known that impact toughness is influenced by many known factors. Not only is heat treatment a critical parameter, but also the amount of Sr, Fe, Mg, grain size, defects and microstructure [18-23]. In this case it is clear that only the grain size does not explains the low impact toughness but defects such as small voids or porosity does.

Even though P/M+HIP aluminum and the AA1100 aluminum cold rolling plate showed dimples in the fracture surfaces, they clearly indicated differences in crack growth between them. This probed by the presence of pores and inclusions on the fracture surface of the P/M+HIP aluminum, while there is no existence of such defects in the fracture surface of the AA1100 aluminum cold rolling plate. This fact indicates that defects played an important role in the crack growth.

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Conclusions

The previously discussed results led to the following conclusions.

It is possible to fabricate billets using P/M+HIP starting from aluminum powders but there is the existence of connected porosity, which has an effect in the mechanical integrity of the billet.

This result was evident after the nondestructive inspection, which shows the need of nondestructive testing in component manufactured using P/M +HIP.

The tensile testing showed differences in properties between the P/M+HIP and the aluminum plate. The aluminum plate was 80 MPa in UTS, which is equivalent to the maximum material strength determined for sound material with no internal defects. Meanwhile, the P//M+HIP sample was around 100 MPa.

The impact toughness, measured using Charpy impact test, of the P/M+HIP billet was much lower than the one of the aluminum plate. This fact indicated a brittle behavior of the P/M +HIP material.

Defects such as pores or inclusions play an important role in the crack growth of P/M+HIP aluminum as it was seen in the fracture surfaces of the tensile and Charpy test samples.

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