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Metallurgical Characterization of Aluminum Alloys by Matrix Dissolution

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Abstract

Aluminum foils produced by cold rolling are able to show defects caused by primary particles that are formed along center line segregation. These particles are formed during casting process where some parameters are critical. In the same way, aluminum extruded parts are also able to show defects caused by intermetallic phases. They were used billets of AA6351 alloy (Al-Mg-Si) produced by "Direct Chill" and also sheets of AA8011 alloy (Al-Fe-Si) produced by "Twin Roll Casting". Samples were submitted to a chemical etching based on methanol and iodine whose target is corrodes only aluminum matrix. Thus, intermetallic phases were analyzed using optical microscopy and SEM and EDS. Beyond, X-Ray Diffraction analysis were performed as well. On AA8011 alloy, they were identified the phases β-$FeSiAl_5$ and α- Fe_2SiAl_8 in as-cast samples, that are hazardous to final foil because they do not dissolve during homogenization treatment. On AA6351 alloy, they were identified the phases Mg_2Si and α-$(Fe, Mn)_3Si_2Al_{15}$. The identification of these phases contributed to process optimization.

Introduction

Aluminum foils with thickness lower than 200μm have large applications as automotive thins, container foil, peel-off, white cap, yogurt, house and hold foil, conversion foil and others. In these cases, aluminum is used due to its high corrosion resistance, impermeability to humidity and oxygen, besides, there is the workability that favors processes as rolling, bending, cutting generating opportunities to transformation industries [1]. The raw material to produces these foils can be produced by DC casting or Twin Roll Casting in which the material is produced with thickness from 3 to 7mm. In worldwide outlook, this process produces AA1XXX, AA3XXX, AA5XXX, AA7XXX and AA8XXX alloys [2, 4].

In the other hand, there are disadvantages among which the. This kind of segregation is also named center line segregation and it is formed in the half of the thickness and its formation is linked to the casting speed. Its formation mechanism is associated to the shear of the rolls upon the first dendrites created during solidification pushing liquid phase rich in solute that solidifies.

The AA8011 alloy is widely used on thin foil production for container foil. When they are cast by TRC process they usually show a center line segregation composed by eutectic lamellar Al-Fe-Si. These intermetallic particles are harder than aluminum matrix and if the thermo mechanical processing was not suitable these particles can affect the mechanical properties of the final foil generating holes and high porosity [3, 5].

In other application, there are the extruded shapes used on building, frames, automotive and aerospace industry. The increasing of these products demand has resulted in requirements to research and development. On AA6XXX alloys, a great amount of intermetallic phases are formed during the solidification, homogenization and heat treatment. [9]

The formation of intermetallic phases is influenced by chemical composition and cooling rate during the solidification. And when they are not dissolved properly during the heat treatment or due to any process failure, scratches, cracks and reduction of fatigue resistance can lead to a failure. [10]

The conventional process used to billets production is the DC casting that consists in pouring the aluminum alloy previously prepared, in other words, after the addiction of grain refiner, degassing and filtering the metal goes through the molds with Airslip® technology. In conventional casting processes there is the formation of shell zone that is a layer composed by coarse grains and oxides formed in the billet surface that is harmful to the further process, extrusion. Using the Airslip® technology, metal does not have contact with the mold, but only with thin film composed by air and lubricant reducing its formation and the inverse segregation. [11]

In order to obtain an alloy with proper microstructure not only the casting parameters are important but also the homogenizing treatment of the as cast material. An ideal homogenizing treatment eliminates the microsegregation, changes the morphology of the insoluble particles and precipitates on aluminum matrix as fine dispersion of Mg_2Si.

The characterization of intermetallics by conventional optical microscopy is not as accurate as a SEM analysis. For both, a chemical etch that dissolves only the aluminum matrix and keep the intermetallics intact is necessary as well as an analysis involving X-ray diffraction. Some authors, [6, 7, 8, 12, and 13] showed analysis performed using this technique and that allows a complex analysis of these phases using matrix dissolution.

The aim of this work is developing a method to dissolves the aluminum matrix using Al-Fe-Si and Al-Mg-Si alloys produced by DC and TRC in order to promotes an improvement of the process parameters and thus improve the product quality

Experimental procedure

To this work, they were used AA8011 and AA6351 alloys, in which AA8011 was produced by TRC with thickness of 7 mm and 2000 mm of width according to the following chemical composition:

Table I. Chemical composition of AA8011 alloy according to ASTM (% weight)

Alloy	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti
AA8011	0,5-0,9	0,6-1,0	0,10	0,20	0,05	0,05	0,10	0,08

The AA6351 alloy was produced by DC with billets of 8" of diameter according to the following chemical composition:

Table II. Chemical composition of AA6351 alloy according to ASTM (% weight)

Alloy	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti
AA6351	0,7-1,3	0,5	0,1	0,4-0,8	0,4-0,8	-	0,20	0,2

For macrostructural characterization (figures 1 and 2) samples were CUT and etched using HBF_4 1,8%, by 3 minutes, showing the typical structure of sheets produced by TRC with fine grains in the edges, according to figure. Microstructural analyses were performed using a microscope Olympus and etched with Keller's reagent (5ml HNO_3, 5ml HCl and H_2O) by 10s.

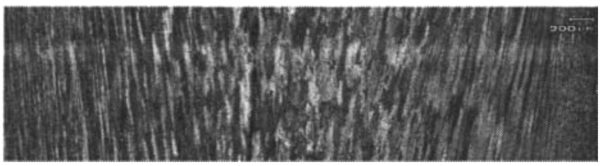


Figure 1. Macrostructure of AA8011 alloy as-cast showing the center line segregation (thickness= 7 mm). Etch (HBF_4 1,8%).

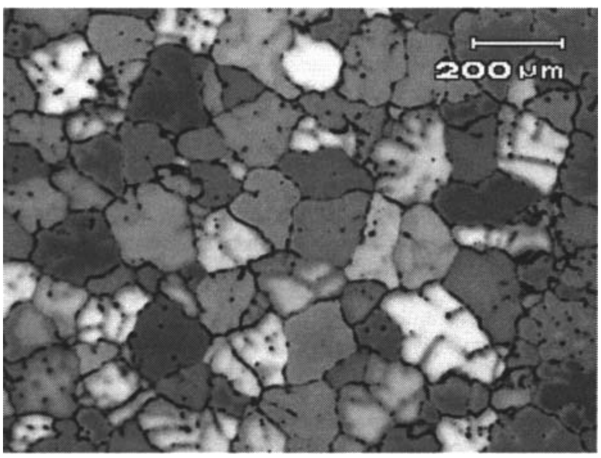


Figure 2. Macrostructure of AA6351 alloy as-cast. Etch (HBF_4 1,8%).

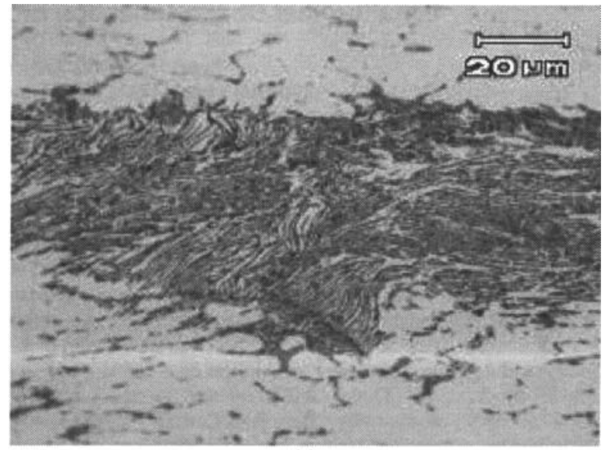
Samples were cut with dimensions of 1cm^3 in order to dissolves the aluminum matrix. Samples were etched using a Becker containing a solution of iodine plus acetic acid (1:3) with methanol (10ml) for 12 hours and then analyzed by SEM to identify the particles morphology. After that, the analysis using EDS was performed in order to identify the phases.

The characterization of phases was performed using X-ray diffraction, as well. To this work it was used a model PANalytycal Xpert PRO.

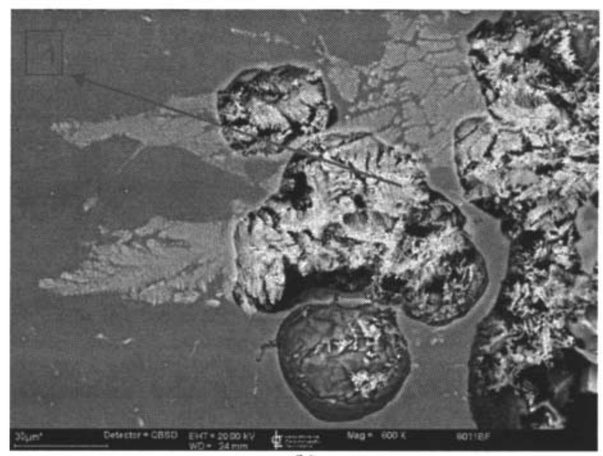
Results and Discussions

Al-Fe-Si Alloy

Figure 3 shows in (a) the microstructure in optical microscope and (b) SEM after the partial matrix dissolution. In both pictures, it is possible to see a lamellar eutectic Al-Fe-Si phase. In (c) there is the result of EDS analysis performed at point 1, as showed at figure 3b. This phase does not show harmful effect on aluminum foil because it is dissolved during homogenizing treatment.



(a)



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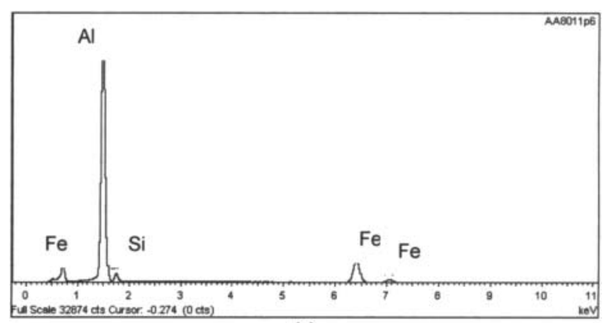
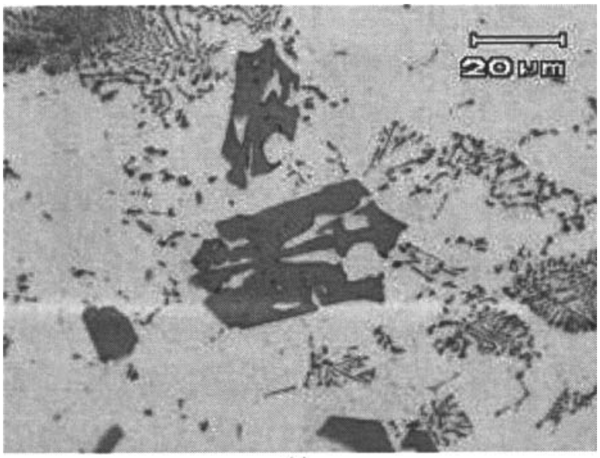


(c)

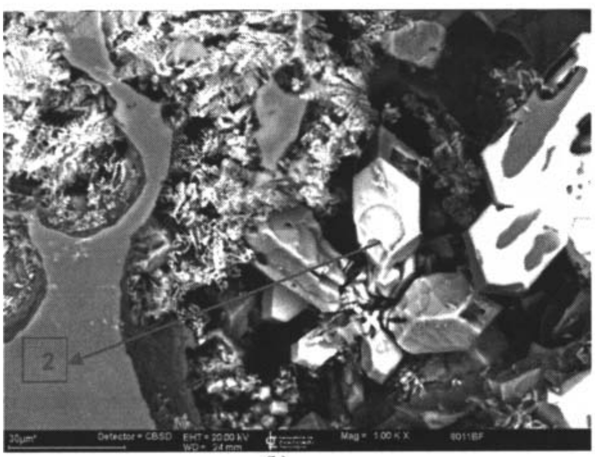
Figure 3. Images of AA8011 alloy under optical microscopy (a) and SEM (b) and the results of EDS analysis at point 1 (c).

Figure 4 shows images of other intermetallics analyzed by optical microscopy (a) and SEM (b) after partial matrix dissolution and (c) after total matrix dissolution in which intermetallics were isolated and analyzed. It is observed the presence of primary particles rich on Al-Fe-Si as observed by

EDS analysis on figure (d). Figures 4 (a) and (b) also show an eutectic lamellar phase involving the primary particles.



(a)



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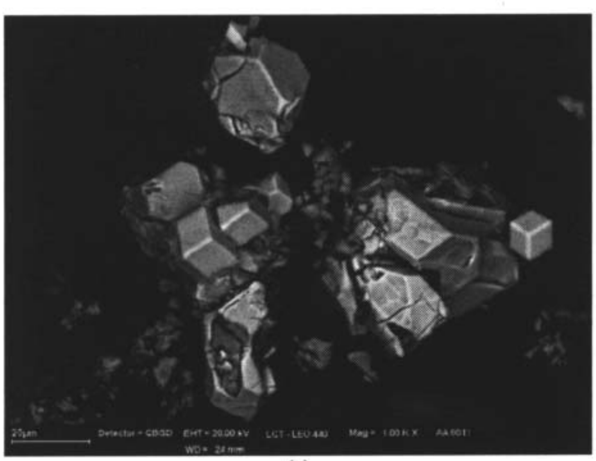

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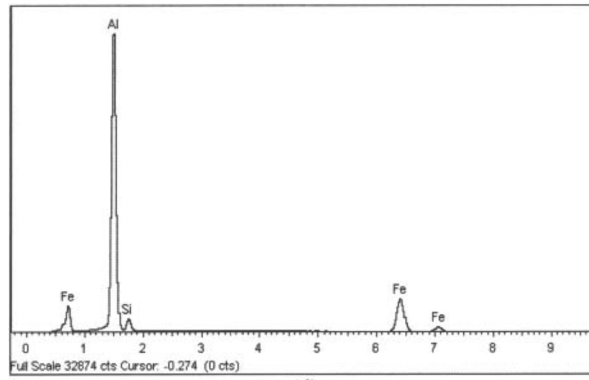


(d)

Figure 4. Images of AA8011 alloy under optical microscopy (a) and SEM (b and c) and the results of EDS analysis at point 2 (d).

These particles promote a harmful effect to the aluminum foil because they do not dissolve during the homogenizing treatment.

X-ray diffraction analysis show the phases on AA8011 as-cast, figure 5. It was possible to identify β-$FeSiAl_5$ and Fe_2SiAl_8 phases, as related for other authors and is related to isolated particles after partial dissolution. In the other hand, it was not possible proves the exact composition for each phase due to difficulty of separation.

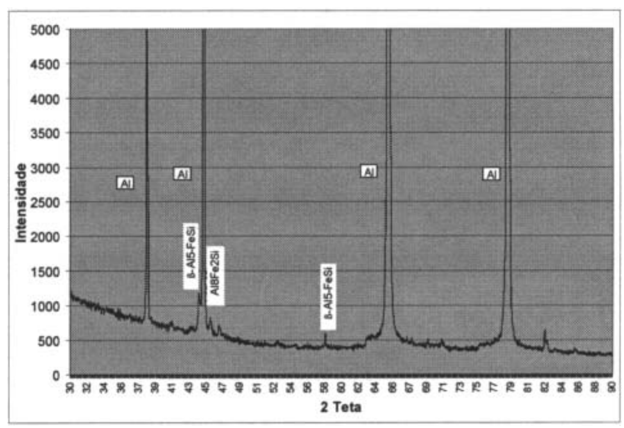
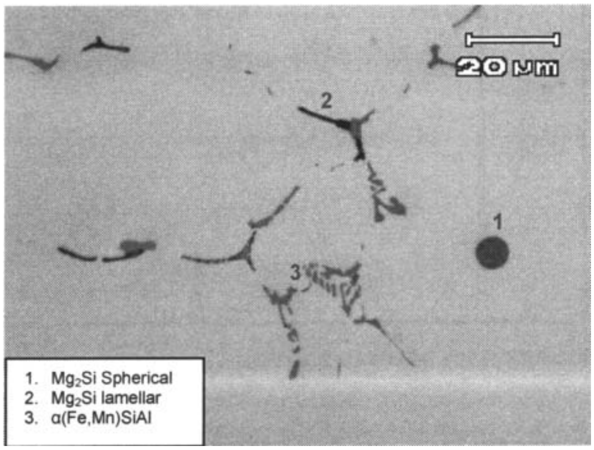


Figure 5. X-Ray Diffraction of AA8011 alloy as-cast.

<u>Al-Mg-Si Alloy</u>

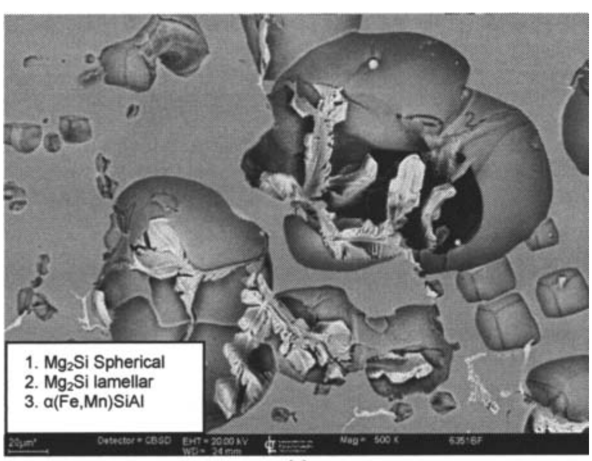
Figure 6 shows in (a) an image of AA6351 alloy as-cast by optical microscopy. It is possible to observe Mg_2Si spherical (1), Mg_2Si lamellar (2), and α(Fe,Mn)SiAl (3) phases. Phases showed in (1) and (2), do not show harmful effect to the final product because they dissolve during homogenizing treatment. Phase showed in (3) does not dissolve during this heat treatment.



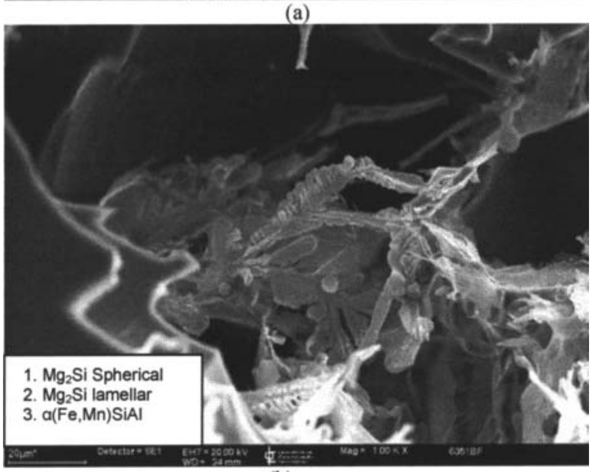
(a)

Figure 6. Images of AA6351 alloy as-cast under optical microscopy.

Figure 7 (a) shows the AA6351 alloy as-cast by SEM after the partial matrix dissolution in which, the same phases of the figure 6 (a) can be seen in 3D.



(a)



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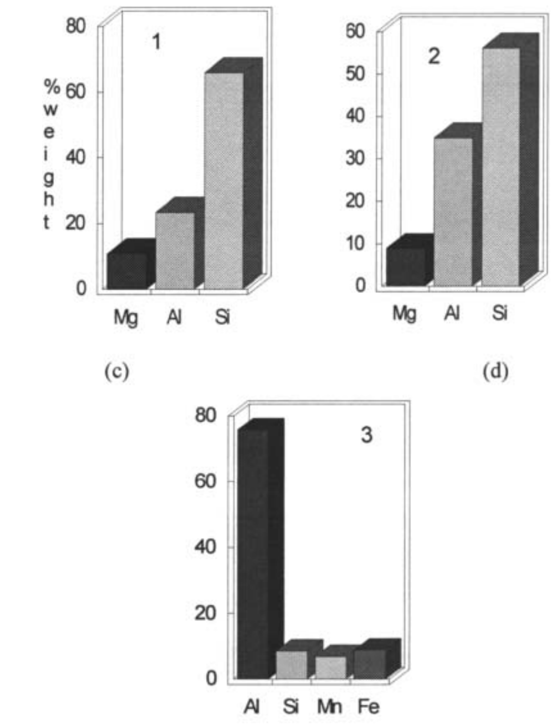


(c) (d)

(e)

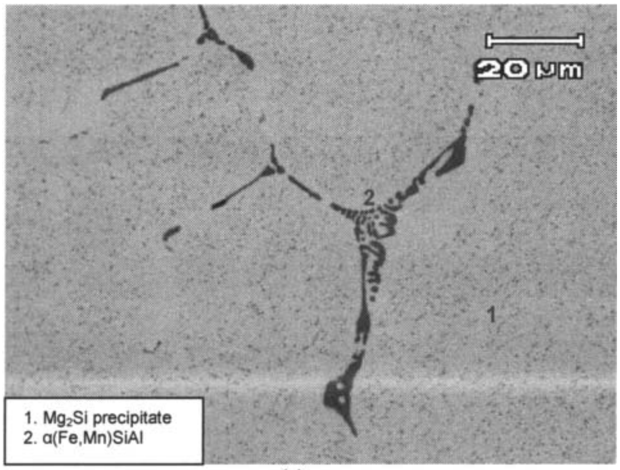
Figure 7. Images of AA6351 alloy as-cast by SEM. (a) Magnification 500X and (b) Magnification 1000X. Spectrum of chemical composition by EDS (c), (d) and (e) at points 1, 2 and 3.

In figure 7 (b) it possible to see the three different phases clearly. At point 1, Mg_2Si spherical, at point 2, along the grain boundary, Mg_2Si lamellar and at point 3, α(Fe,Mn)SiAl. The figures 7 (c), (d) and (e) show the chemical composition of the analyzed phases. The result observed in (c) and (d) prove the formation of Mg_2Si and (e) the α(Fe,Mn)SiAl phase.

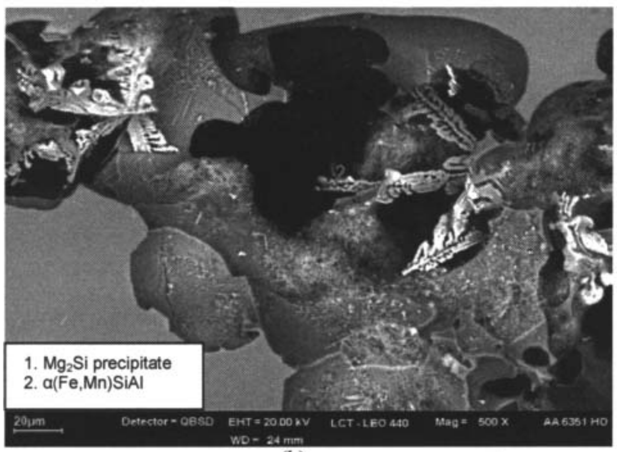
Homogenized samples can be seen at figure 8. At image (a), it can be seen the microstructure under optical microscopy. It is possible to see the α phase with secondary arms coalesced (point 2) and the absence of Mg_2Si along the grain boundaries and at spherical morphology, that suggests what is expected for this alloy, in which this phase precipitate at aluminum matrix as point 1.

The results of EDS are showed at images (c) and (d).

The X-ray diffraction shows the phases at AA6351 alloy as-cast, figure 9. It was possible to identify Mg_2Si and α-(Fe, Mn)$_3$SiAl$_{15}$ alloy, as related by others authors.



(a)



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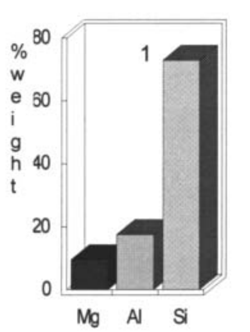

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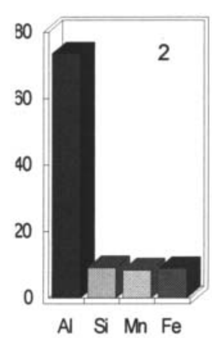

(d)

Figura 8. Images of AA6351 homogenized and analyzed by optical microscopy. (a) 500X magnification and SEM (b) 500X magnification. Spectrum of chemical composition by EDS (c), (d) at points 1 and 2.

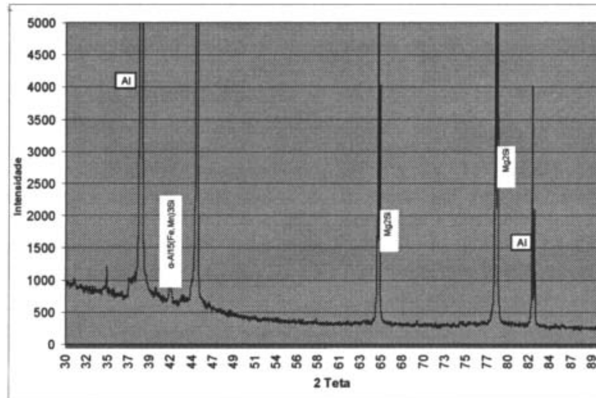

Figure 9. X-Ray Diffraction of AA6351 alloy as-cast.

Conclusions

It was possible to reach the aim of this work in which the aluminum matrix was dissolved keeping intermetallics intact in order to determine their chemical composition and morphology. Using AA8011 alloy they were identified the phases β-$FeSiAl_5$ and Fe_2SiAl_8 however, it was not possible identify the correct chemical composition for each particle due to difficulty of separation. Using AA6351 they were identified α-$(Fe,Mn)_3SiAl_{15}$ and Mg_2Si phases as spherical and lamellar morphology in as cast samples and only spherical morphology after precipitation phase. A future opportunity to this work is using potassium ferricyanide plus sodium hydroxide as chemical etching. This method will be tested and published further.

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