Light Metals 2012 *Edited by: Carlos E. Suarez*
TMS (The Minerals, Metals & Materials Society), 2012

ANALYSIS OF THERMAL AND STRUCTURAL PARAMETERS AND MICROHARDESS VARIATIONS IN DIFFERENT AL-CU ALLOYS DIRECTIONALLY SOLIDIFIED

Carlos M. Rodriguez[1,2]; Adriana E. Candia[1]; Carlos E.Schvezov[1,3]; Mario R. Rosenberger[1,3]; Alicia Ares[1,3]

[1] Materials, Modeling and Metrology Program, Faculty of Sciences, University of Misiones, 1552 Azara Street, 3300 Posadas, Argentina.
[2] Doctoral Fellow of the National Science Research Council of Argentina (CONICET). 1917 Rivadavia Street, 1033, Bs. As., Argentina.
[3] Member of CIC of the CONICET, Argentina. 1917 Rivadavia Street, 1033, Buenos Aires, Argentina. E-mail: aares@fceqyn.unam.edu.ar.
[4] Chairman of the Board of Development and Technological Innovation (CEDIT), 1890 Azara Street, 5th Floor, 3300 Posadas, Argentina.

Keywords: Al-Cu alloys, directional solidification, thermal parameters, structural parameters, mechanical properties.

Abstract

The columnar - to - equiaxed transition (CET) was investigated in Al-Cu alloys (Al-1wt%Cu, Al-4.5wt%Cu, Al-15wt%Cu and Al-33.2wt%Cu) solidified directionally from a chill face in a vertical setup. The CET occurs when the temperature gradient in the melt ahead of the columnar front reach critical values. Also, we investigate correlations between structural parameters (grain size) with Vickers microhardness measurements in the directionally solidified samples. We observed that the Vickers microhardness is greater in the equiaxed zone than in the columnar or columnar to equiaxed transition (CET) zone, additionally, is greater on the edges of the samples than in the centre. Also, we determined that the grain size increases from the columnar to equiaxed structure.

Introduction

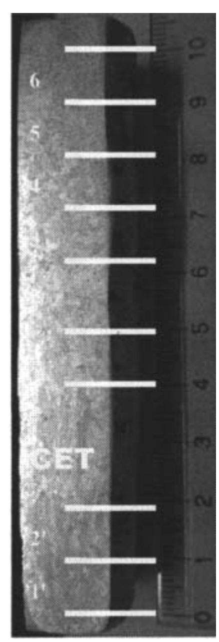
The results presented in this report are the continuation of previous investigation on the columnar to equiaxed transition in others alloy systems [1-3] which are now expanded to other alloys of technological interest like the aluminum-copper alloys. In the previous investigations the effect of several solidification parameters on the transition was determined and discussed. Such parameters include the position of the solidification fronts given by the liquidus and solidus temperatures, cooling velocity of the liquid and temperature gradient. The alloys selected for the present study are Al-1wt%Cu, Al-4.5wt%Cu, Al-15wt%Cu and Al-33.2%wtCu [4].

Materials and Methods

The alloys were solidified directionally upwards in an experimental set up consisting of a heating unit, a temperature control system, a temperature data acquisition system, a sample moving system and a heat extraction system. The set up is shown in Figure 1. After solidification the samples were cut in the axial direction, the samples were polished and etched with a solution consisting of 15ml HF, 4.5ml HNO_3, 9ml HCl, 271.5ml H_2O, in the case of the alloys with less than 10%wtCu. A solution of 320ml HCl, 160ml HNO_3, 20ml HF was utilized as etching for alloys with more than 10%wtCu [5]. The position of the transition was determined by observation under an optical microscope.

The equiaxed grain size was measured using the ASTM E112 standard norm [6], at equally spaced intervals. The columnar region was divided in similar way and the width and length of the grains measured directly. In Figure 2 it is shown the standard distribution of intervals used for a typical sample.

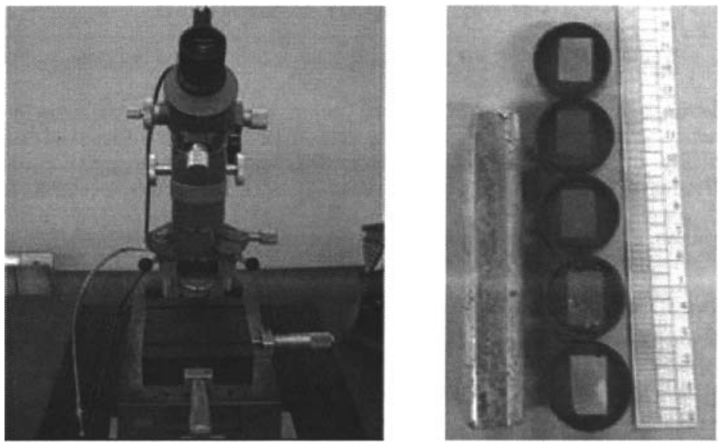
Figure 1. Experimental setup.



Scale 0.8 : 1

Figure 2. Al-33.2wt%Cu alloy sample.

Microhardness measurements were performed at room temperature with a Leitz Durimet ® microhardness tester (see Figure 3). Loads between 50 g were used. The measurements were performed under ASTM E 384-89 [6] standard using a pressing time of 15 seconds.



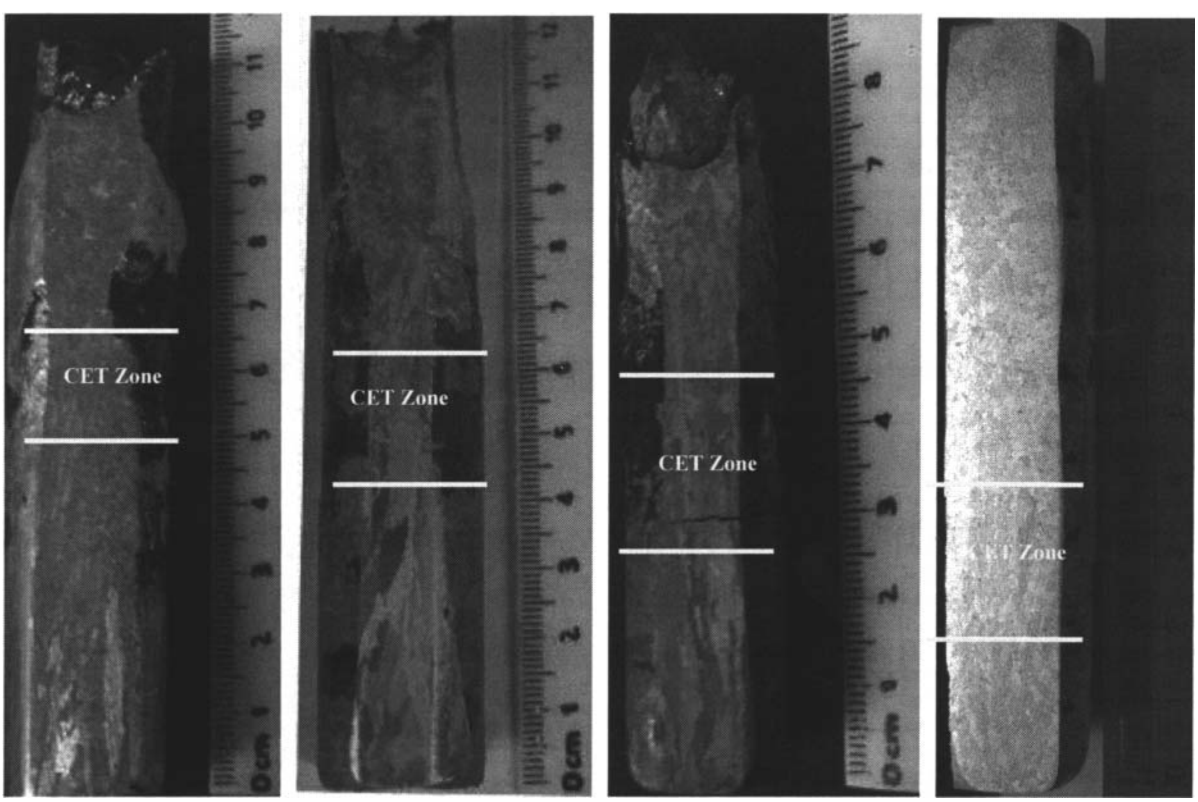
(a) (b)

Figure 3. (a) Vickers Microhardness (Leitz Durimet) Laboratorio de Metalografia –CNEA. Buenos Aires. Argentina. (b) Alloys samples.

Results and Discussion

Columnar-to-Equiaxed Transition

A number of 22 experiments were performed where the transition from columnar to equiaxed structure was produced. Typical columnar – to – equiaxed transitions can be observed in Figure 4 for the different alloys tested, Al commercial, Al-1%wtCu, Al-33.2%wtCu, Al-5%wtCu. In Figures 4 it is clearly seen that the transition do not occur sharply but in a region of 1cm or more.



(a) Scale 0.9 : 1 (b) Scale 1 : 1 (c) Scale 1.2 : 1 (d) Scale 1 : 1

Figure 4. The CET of different Al-Cu alloys. (a) Al-1%wtCu. (b) Al-4.5wt%Cu. (c) Al-15wt%Cu. (d) Al-33.2%wtCu.

Thermal Parameters

The cooling velocity of the liquid alloy was determined from the temperature versus time curves at each thermocouple position and taking the average slope.

The temperature versus time for the experiences corresponding to alloys Al-1wt%Cu, Al-4.5wt%Cu, Al-15wt%Cu and Al-33.2%wtCu are presented in Figure 5. The cooling velocities calculated from these types of curves are listed in Table I for all the experiments as $V.E._{LIQ.}$; velocities of 0.23 to 0.69 °C/s were produced. Table I also lists the location of the transition from the bottom of the sample which is in the range of $CET_{MIN.}$ to $CET_{MAX.}$ (cm). Comparing the cooling velocities with the distances which correspond to the length of the columnar zone, for Al-Cu alloys, it is observed that increasing the velocity increases the length of the columnar grains.

The temperature versus time curves also show that the temperature evolution depends on the structure being formed. During columnar solidification the temperature decreases steadily and monotonically, on the contrary in the equiaxed region, during the transition, there is a recalescence which increases the temperature from a minimum; the level of recalescence for each experiment is listed in Table I as REC (°C).

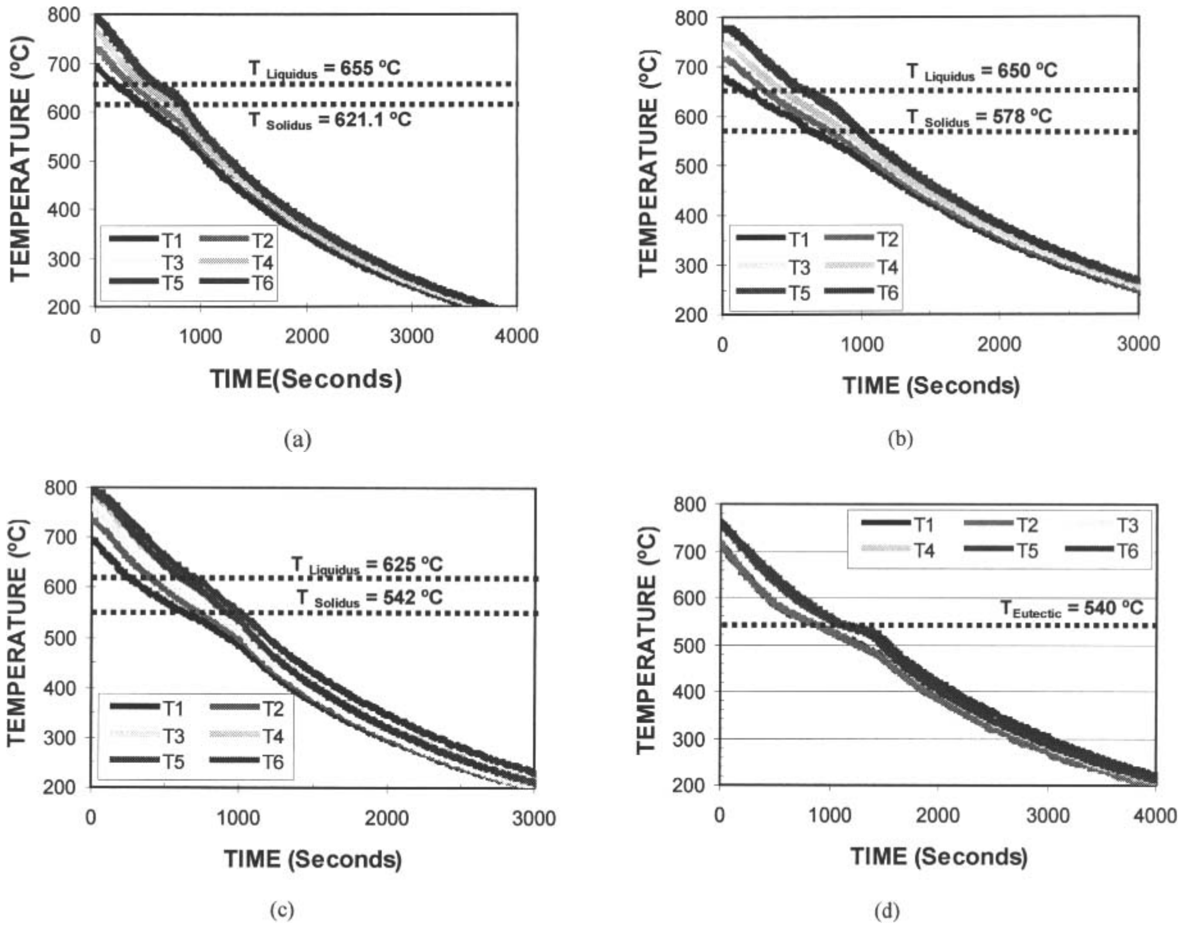


Figure 5. Cooling curves. (a).Al-1wt%Cu. (b) Al-4.5%wtCu. (c) Al-15%wtCu. (d) Al-33.2%wtCu.

The temperature gradients, G, were calculated for each pair of neighbor thermocouples as the temperature difference between the thermocouple readings divided by the separation distance between thermocouples. According to this the error in the calculated gradients is

$$\Delta G = \frac{G}{T_{i-1} - T_i} + 0.04.G \qquad (1)$$

The values of gradients are plotted in Figure 6 (a to d) for three experiments, and for each alloy. In all the figures it is observed that from the beginning of solidification, the gradients decrease with time. The minimum value always corresponds to the position of the columnar to equiaxed transition. The gradients shown in Figure 6 (c) reach negative values at this point, which is –4.02 °C/cm. This negative value is an indication of a reversal in the temperatures profiles ahead of the interface, which could be associated to the recalescence due to massive nucleation of equiaxed grains, and previously reported and discussed for other alloys [1-3]. The fact that in some cases the position of the thermocouples are not located at the precise position where the transition occurs, may prevent detection of the negative gradients which is believe to occur in all cases. Nevertheless, the values always reach a minimum value at this position which, in the case of Figures 6 (a), (b) and (c) are of 0.67 °C/cm, 1.33 °C/cm and 3°C/cm. The values of 5 to 30 °C/cm are observed at the beginning of solidification.

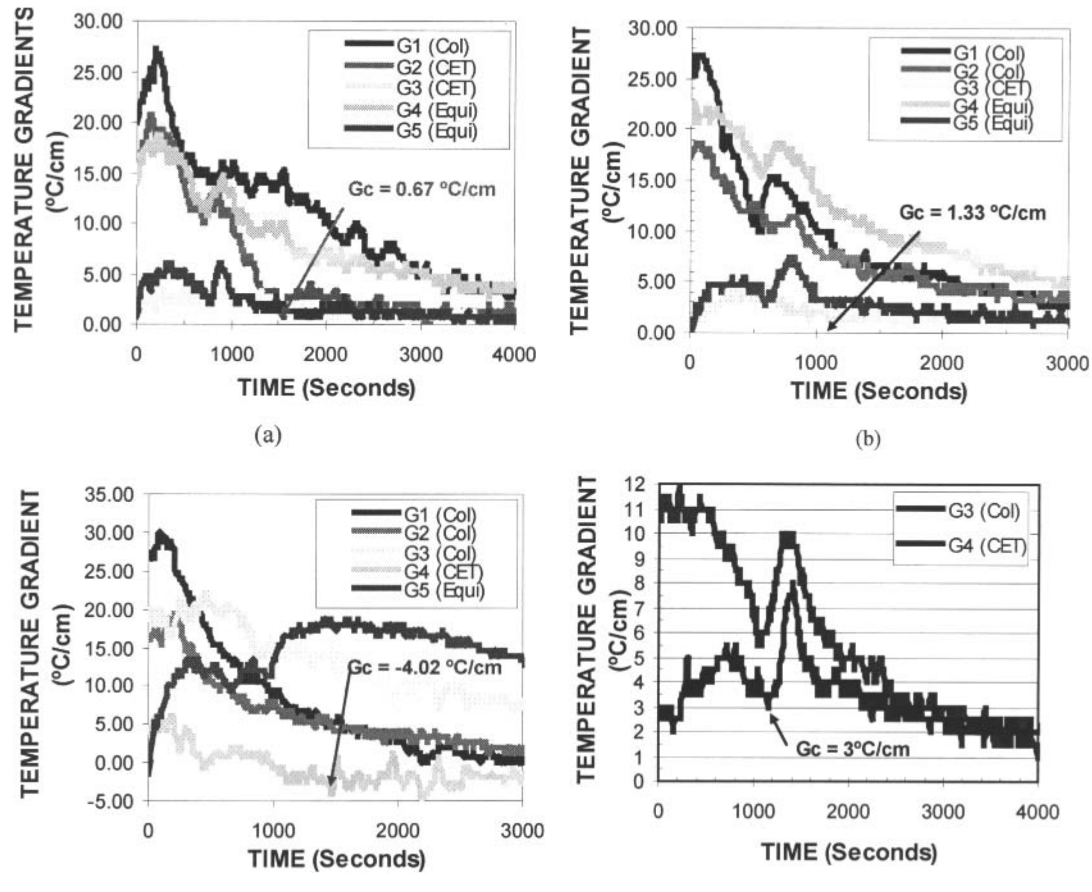


Figure 6. Temperature gradient versus time. (a).Al-1wt%Cu. (b) Al-4.5%wtCu. (c) Al-15%wtCu. (d) Al-33.2%wtCu.

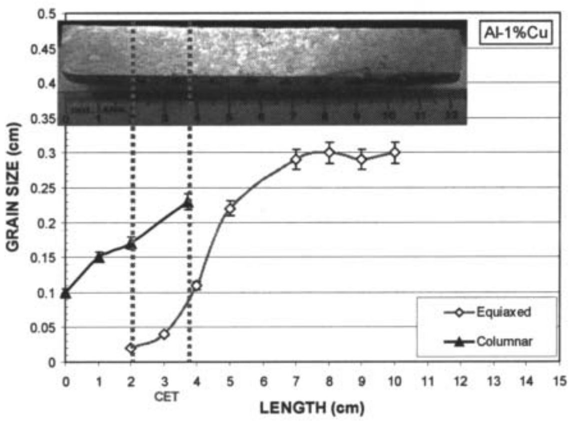
Table I. Liquidus temperature (T_L), solidus temperature (T_S), cooling velocity of the liquid (V.E.$_{LIQ.}$) and cooling velocity of the solid (V.E.$_{SOL.}$), minimum CET position (CET$_{MIN}$.) and maximum CET position (CET$_{MAX}$.), critical temperature gradients (G_C) and recalescence values (REC.) obtained from the temperature versus time curves.

N°	ALLOY	T_L (°C)	T_S (°C)	V.E.$_{LIQ.}$ (°C/s)	V.E.$_{SOL.}$ (°C/s)	CET$_{MIN}$ (cm)	CET$_{MAX}$ (cm)	G_C (°C/ cm)	REC. (°C)
1	Al-1wt%Cu	655	621	0.69	0.17	6.5	5.0	0.67	0.69
2	Al-4.5%wtCu	650	578	0.36	0.22	6.3	4.2	1.33	0.28
3	Al-15%wtCu	625	542	0.33	0.26	4.6	2.5	-4.02	0.42
4	Al-33.2%wtCu	540	540	0.23	0.17	4.0	1.8	3.0	1.02

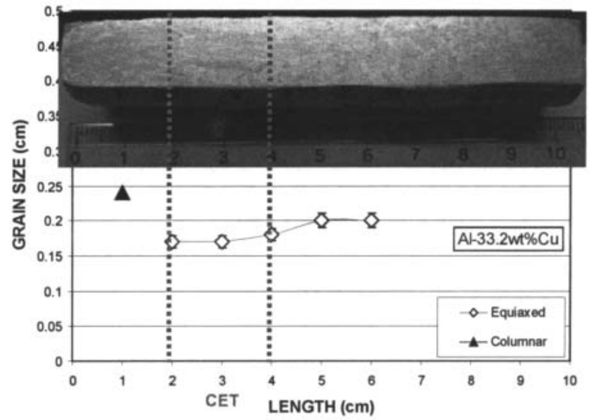
Grain Size Measurements

From a typical histogram showing the frequency of the size of the equiaxed grains the equiaxed grain size was determined. The results, which also include the width of the columnar grain, are plotted as function of position in the solidified sample for two samples in Figure 7.

In Figure 7 (a), the size of the equiaxed grains is 0.04 cm in the transition region and then starts to monotonically increase to a value of 0.3 cm at the end of the sample, which is the part of the alloy that solidified last. In the case of the width of the columnar grains it is observed that they increase in size when approaching the transition region. Similar were performed for a total of solidification experiments. In Figures 7 only two of them are presented, corresponding to Al-1wt%Cu and Al-33.2%wtCu alloys. The points in the figures were fitted to polynomial functions of third degree. In Figure 7, it is observed that in the transition, the size of the equiaxed grains is smaller than the width of the columnar grains. However, in all the cases, the size increases after the transition. At the end of solidification, the size may also decrease after reaching a maximum value. This behavior was previously reported in our experiments [1-3] and from micrographs published by Lowe, Mahapatra, Biloni and Gandin and Rappaz [1] for different alloy systems. Another observation that is common to all the cases presented here is that the dependence of the equiaxed grain size with position from the transition is independent of position of the transition, i.e. length of the columnar region, alloy composition or relative size of the equiaxed grains.



(a)



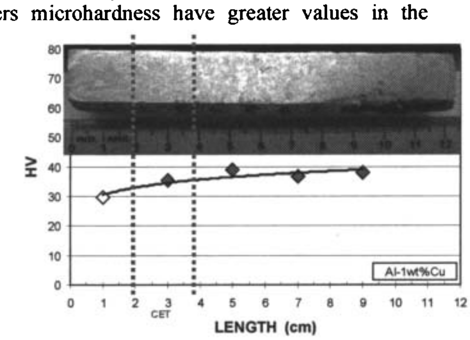
(b)

Figure 7. Grain size evolution versus length of the sample. (a) Al-1wt%Cu. (b) Al-33.2wt%Cu.

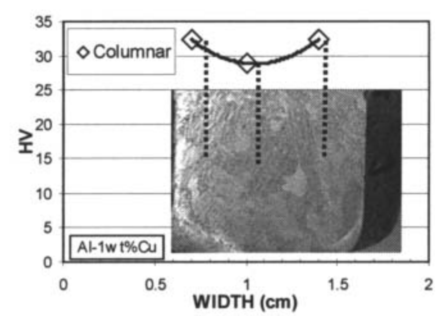
__Microhardness Measurements__

To determine an average microhardness a minimum of 10 measurements were performed in each section of the samples (Figures 8). Figures 8 (a) show experimental results of microhardness variations analyses as a function of sample length, for two different alloys, using a load of 50 g_f. It can be seen that Vickers microhardness have greater values in the equiaxed zone of the samples than in the columnar and columnar to equiaxed transition (CET) zones.

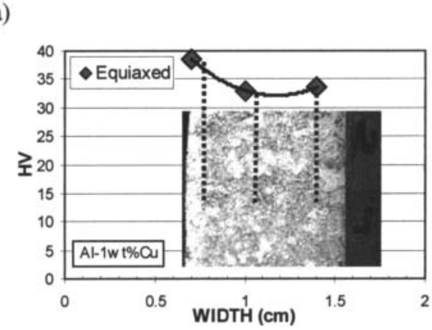
The results of the microhardness variations analysis as a function of sample width are shown in Figures 8 (b) and 8 (c) for different structures. In all cases we obtain greater microhardness values in sample edges than in the centre of the samples.



(a)

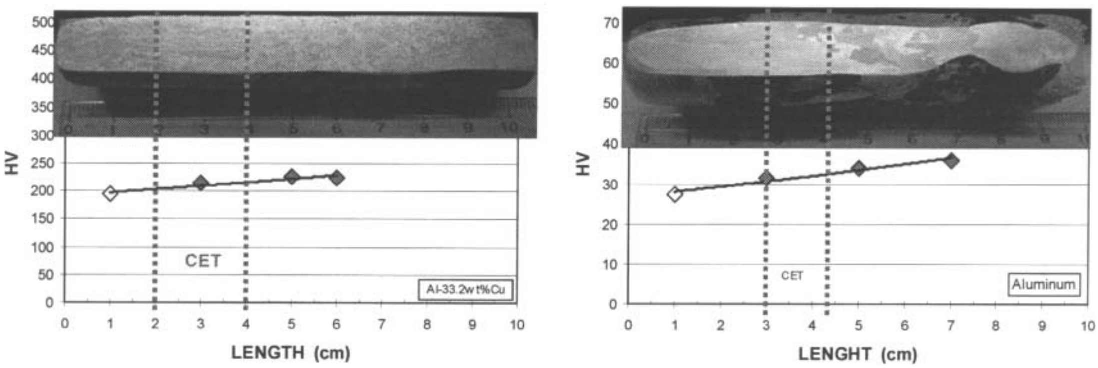


(b)



(c)

Figure 8. Microhardness values versus (a) length and (b) width of the samples utilizing a load of 50 g_f. Al-1wt%Cu.



(a) (b)

Figure 9. Microhardness values versus length. (a) Al-33.2wt%Cu. (b) Commercial Aluminum.

In Figures 9 (a) and (b) it is possible to observe that the Vickers microhardness increases from the columnar zone (bottom of the sample) to the equiaxed zone (top of the sample). Also, that the Vickers microhardness is greater for the eutectic alloy (≅ 200HV) than for the other alloys (≅ 40HV).

Summary and Conclusions

The main conclusions obtained from the present work are as follow:

1) Directional solidification of Aluminum based alloys were performed and the temperatures during the whole process were measured in the liquid and solid phases.
2) For the four type of alloys studied, i.e Al-1wt%Cu, Al-4.5wt%Cu, Al-15wt%Cu and Al-33.2%wtCu, the columnar to equiaxed transition was produced and the values of the temperature gradients, which were calculated, reached minimum values during the transition; in some cases negative.
3) In the Aluminum-Copper alloys it is reported that an increase in the cooling velocity in the liquid increases the columnar zone length.
4) Recalescence was detected and measured during the equiaxed transition being of the order of 0.28°C to 1.02°C.
5) The transition from columnar to equiaxed structure is not abrupt but occurs in a zone of 1 cm or larger.
6) The equiaxed grains have size distributions that have a time and position evolution, which is practically independent of the type of alloy, concentration and size of the grains, giving small grains at the transition and increasing ahead of the transition.
7) The Vickers microhardness is greater in the equiaxed zone than in the columnar or columnar to equiaxed transition (CET).
8) The Vickers microhardness is greater on the edges of the samples than in the centre.
9) The Vickers microhardness is greater for the eutectic alloy (≅ 200 HV) than for the other alloys (≅ 35 HV).

Acknowledgements

This work was supported by PIP 2010-2012 GI 11220090100769 from the Argentinean Research Council (CONICET).

References

1. A.E. Ares, and C.E. Schvezov, Solidification Parameters during the Columnar-to-Equiaxed Transition in Lead-Tin Alloys, Metall. Trans., 31A, (2000), 1611-1625.

2. A.E. Ares, and C.E. Schvezov, Influence of Solidification Thermal Parameters on the Columnar-to-Equiaxed Transition of Al-Zn and Zn-Al Alloys, Metall. Trans., 38 A, (2007), 1485-1499.

3. A. E. Ares, S. F. Gueijman, C. E. Schvezov, An Experimental Investigation of the Columnar-to-Equiaxed Grain Transition in Aluminum-Copper Hypoeutectic and Eutectic Alloys, Journal of Crystal Growth,, 312, (2010), 2154–1170.

4. W.J. Moffatt, Handbook of Binary Phase Diagrams, Published by General Electric Company Corporate Research and Development Technology Marketing Operation, New York, (1984), 259, 419, 437, 391.

5. G. Kehl, Fundamentos de la Práctica Metalográfica, Editorial Aguilar, Madrid (1963).

6. H. E. Boyer and T. L.Gall, Metals Handbook, Desk Edition. American Society for Metals, (1990) 35-18. 35-19.