N.V. Kawecki-Billiton Metaalindustrie

TITLE: INTERRELATIONS BETWEEN ALUMINIUM GPAIN REFINING BY MEANS OF ALUMINIUM TITANIUM BORON ALLOYS AND THE NUMBER OF GROWTH CENTRES.

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Over the past decades grain refining by means of AlTiB alloys has become common practise. Due to its effectivity, the application of AlTi5B1 became most widespread. In the recent past the emphasis was laid on the cleanliness of AlTiB alloys rather than the effectivity. Consequently, the requirements of the aluminium industry have necessitated the development of other Ti/B ratio's. Nowadays, the cleanliness of these particular alloys has been significantly improved by the application of enhanced processing techniques, while simultaneously increasing the effectivity. Therefore, when selecting the optimal grain refiner, allowances should be made for the difference in effectivity. A standard practice to correlate the result of a grain refining test to a grain size in a cast product would simplify the selection process.The system proposed in this paper aims to contribute to such an approach.
The efficiency of a grain refiner can be expressed by grain size. It will be shown, however, that the actual number of persistent growth centres provides a more sensitive parameter for this type of evaluation. The ratio between potential nuclei and actual growth centres depends on several variables such as: alloy composition and cooling rate.
This paper will demonstrate that the AlTi5B1 grain refiner is the most effective.

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Experimental:

Four different grain refining tests were applied to characterize the effectivity of the three most used AlTiB alloys. For our experiments we selected AlTi5B1, AlTi5B0.2 and AlTi3B1 alloys, each being representative for their class of grain refiner.

The following grain refining tests were applied:

1.Kawecki-Billiton ring test	for a description see the appendix
2.Alcan test	according to Alcan specification
3.Reynolds Golf Tee test	according to Reynolds specification
4.V.A.W. test	according to V.A.W. specification

The alloys 99.7% Al., AlMgSi (AA6063) and AlMnMg (AA3004) were used to perform the tests. The alloys were produced from 99.7% Al. through the addition of commercial master alloys.

The grain refining tests were executed by means of addition of 2, 1 and 0.5 kg/ton of the three selected grain refiners to the three Aluminium alloys. All tests were repeated in order to obtain an average figure. The contact time for all tests was 2 minutes.

Grain size was determined by means of the intercept method based on the British Standard DD44: 1975, which is similar to ASTM E112-63.

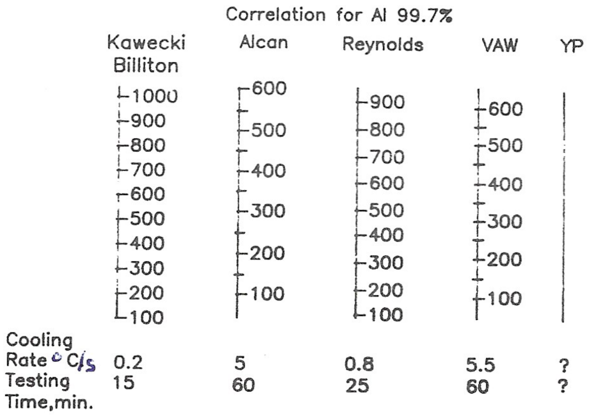
Comparison of grain refining tests

Despite efforts on the part of Kirby et al (ref.1.), aimed at correlating the grain size measured in the grain refining tests to an identical grain size in a cast product, no further attempts were undertaken to obtain an acceptable comparison.

The experiments carried out on the three Aluminium alloys indicated that a reproducable correlation between grain refining tests exists, albeit that the correlation is different for each alloy.

In the case of 99.7% Al., it appears that once the grain size for an addition in one test method is known, the result of any other test can be predicted by using the nomogram as indicated in figure 1. for 99.7% Al.

The Reynolds golf tee test and the Kawecki-Billiton ring test yield very similar results, as also the Alcan test and the V.A.W. test. Therefore, for further correlations on the other two alloys, only the Kawecki-Billiton ring test and the Alcan test were used.
The correlation between the Kawecki-Billiton ring test and the Alcan test for AlMgMn (AA 3004) and AlMgSi (AA 6063) are given seperately in figure 1. It appears that for each alloy the correlation between grain refining tests differs and a univerisal correlation, therefore, does not exist.



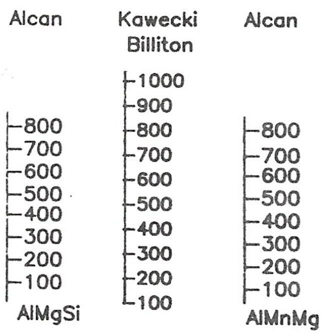


Figure 1.: In the upper section a nomogram to correlate the Kawecki-Billiton ring test, the Alcan test, the Reynolds golf tee test and the V.A.W. test for 99.7% Al. is shown. The grain size is given in microns. The correlation between the Kawecki-Billiton ring test and the Alcan test for AlMnMg (AA 3004) and AlMgSi (AA 6063) is indicated in the lower section. The YP (Your Product) line needs to be established in the casthouse.

Attention should be paid to the fact that a given cast shape (see the Your Product (YP) line in figure 1.) is actually another "system" in which the performance of a grain refiner is tested. The correlation between a grain refining test and the grain size in a cast product can only be established in the casthouse, preferably for each alloy and shape. Once this relationship is established by the user the performance of the grain refiner can be tested prior to its use to establish optimal addition, albeit that allowances should be made for the commercial range of the composition of the aluminium alloy at hand.
It would benefit the aluminium industry should a consensus be reached on which the grain refining test would become the "standard" test. It should be envisaged that for several reasons, a.o. historical preferences, it may prove to be difficult to attain a standard grain refining test. Indeed, nearly all Aluminium companies have their own grain refining test.
To offer a practical alternative, correlations of the type shown in this paper can be worked out for the most commonly used Aluminium alloys and could well result in a kind of standardization.

Grain refiner effectivity

The results of the Kawecki-Billiton ring test (2 minutes contact time at 720 °C.) on the various additions of the three grain refiners are plotted in figure 2.

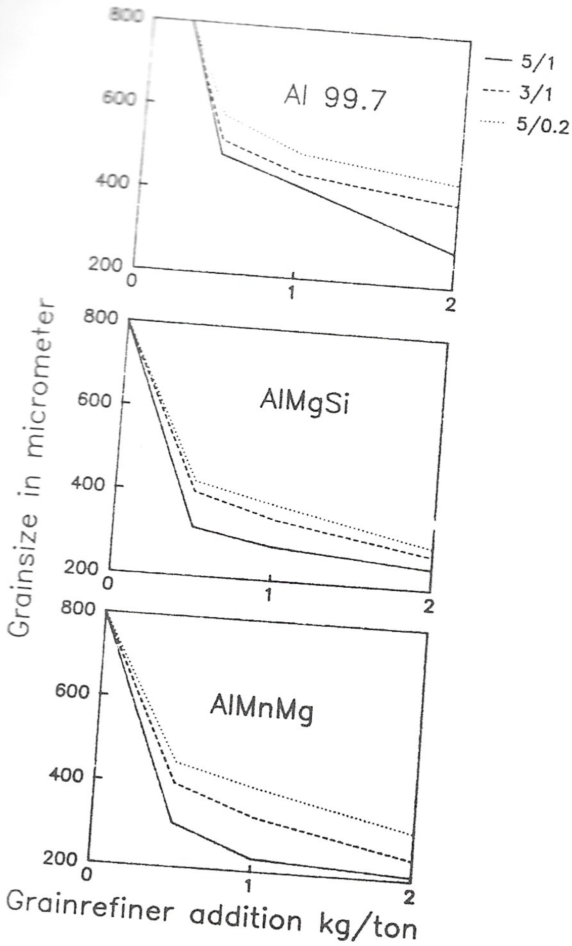


Figure 2.

Grain size in micrometer as a function of addition for the three commercial grain refiners, based on the Kawecki-Billiton ring test

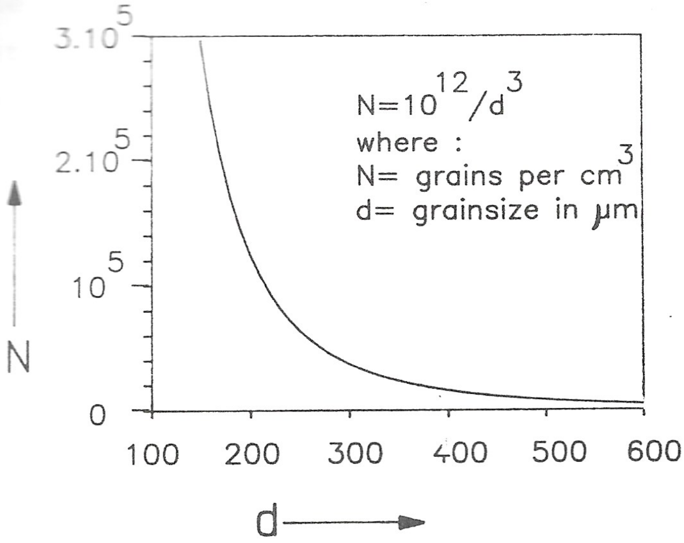
The number of grains (N) per cm^3 can be calculated from the grain size figures (see also the previously mentioned ASTM and British Standard) using the following formulae:

$$N = \frac{10^{12}}{d^3}$$

N = number of grains per cm^3

d = grain size in microns

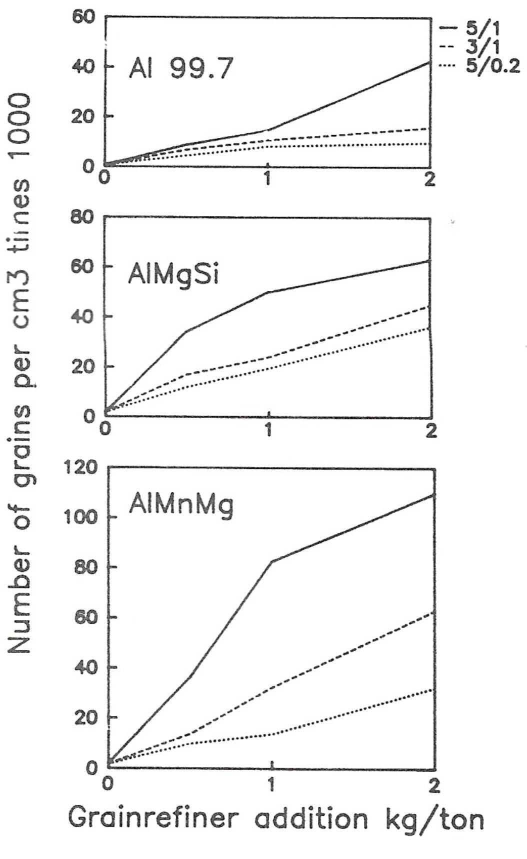
Figure 3. visualizes this relationship



<u>Figure 3.</u>

Relationship between the grain size and the number of grains.

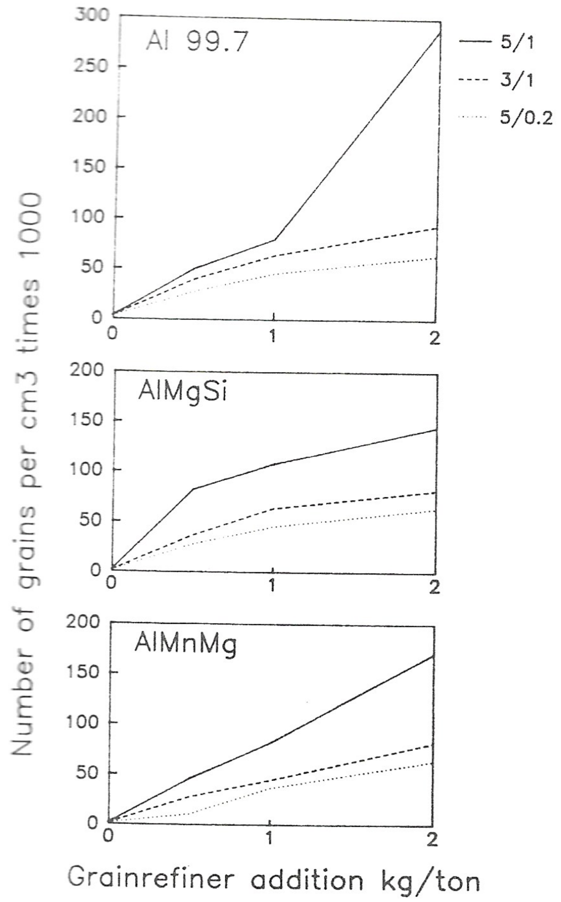
Using this relationship, figure 2. has been replotted to show the relationship between the number of grains and the grain refining addition.



<u>Figure 4.</u>

The number of grains as a function of the addition for the three commercial grain refiners, based on the Kawecki-Billiton ring test (This figure is based on the results of figure 2.)

The same procedure was executed to discover the relationship between the number of grains and the grain refining addition in the Alcan test. The aim being to show the extend of the cooling rate influence on grain refining activity. The results are recorded in figure 5.



<u>Figure 5.</u>

The number of grains as a function of addition for the three commercial grain refiners, based on the Alcan test

On average the AlTi5B1 grain refiner is three times more effective than the AlTi5B0.2 grain refiner and 1.5 times more effective than the AlTi3B1 grain refiner. The effect of residual Titanium or other elements, e.g. Fe etc., were not taken into account in this comparison.
When compared to AlTi5B1, the use of AlTi5B0.2 in pure Aluminium is justified by the reduced Boron content in this alloy rather than by the effectivity. Moreover, the minimum addition of a grain refiner is not limited by grain size but by the necessity to avoid the formation of feather crystals.

The synergetic effect of the alloying and the cooling rate is clear and stresses the need to establish, for each specific case, the previously mentioned correlation between the grain refining test and the grain size in a cast product.

Discussion

As previously has been described by Maxwell and Hellawell (ref. 2.), for a given undercooling the amount of nucleation events as a function of solidification time is shown in figure 6.

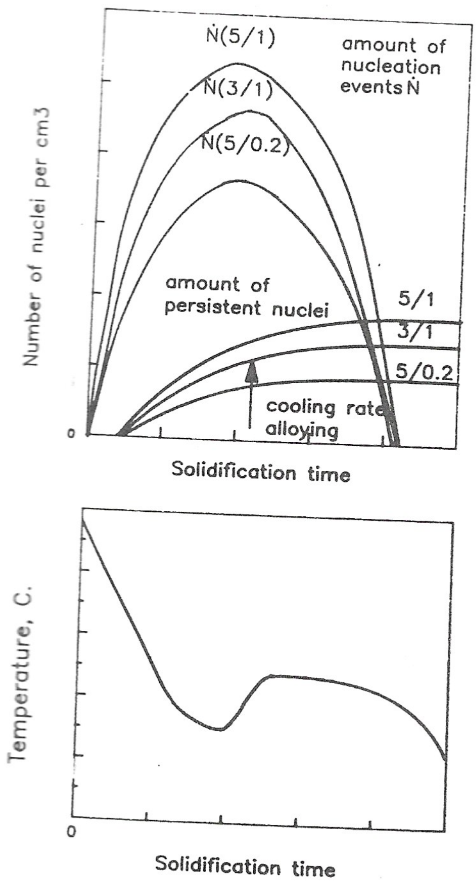


Figure 6.

The relation between the amounts of nucleation events $\dot{N}$ and the number of persistent nuclei N, according to the concept of Maxwell and Hellawel (Ref. 2.) for the three grain refiner alloys.
The course of the cooling curve is indicated in the ~~upper~~ lower graph.

After a certain degree of undercooling, the amount of nucleation events per cm^3 rises rapidly and is at its maximum just before maximum undercooling is reached. It then decreases rapidly as recalescence begins. The amount of persistent nuclei N rises simultaneously with $\dot{N}$ in a more or less fixed ratio, to remain constant as $\dot{N}$ decreases.
The differences between the tested grain refining alloys are also indicated in figure 6. The amount of nucleation events must show a trend similar to that of the difference in effectivity between the three grain refining alloys. The rate of nucleation events increases as a result of alloying and enhanced cooling and, accordingly, more growth centres are activated.
A more satisfactory explanation of the effect of cooling rate and alloy composition can only be found by modelling the local nucleation and growth of sole nuclei, taking into account the interaction with other nuclei. Activities in this field have been initiated.
The expectation from this basic work is to guide the improvement and development of the current AlTiB alloys as well as the non/low Boron grain refining alloys which are currently being developed.

Conclusion

- A better understanding of the efficiency of a grain refiner can be achieved if the amount of grains, rather than the grain size, are taken into account.
- In the case of the three investigated alloys (99.7% Al., AlMgSi [AA6063], AlMnMg [AA3004]), a reproducable correlation exists between the Kawecki-Billiton ring test and the Alcan test (and the Reynolds Golf Tee test and the V.A.W. test for 99.7% Al.), being different for each alloy. When applied to other alloys and grain refining tests, it may prove to be the beginning of a standardization for comparative results of grain refining tests.
- The AlTi5B1 grain refiner is the most efficient commercial grain refining alloy.

References

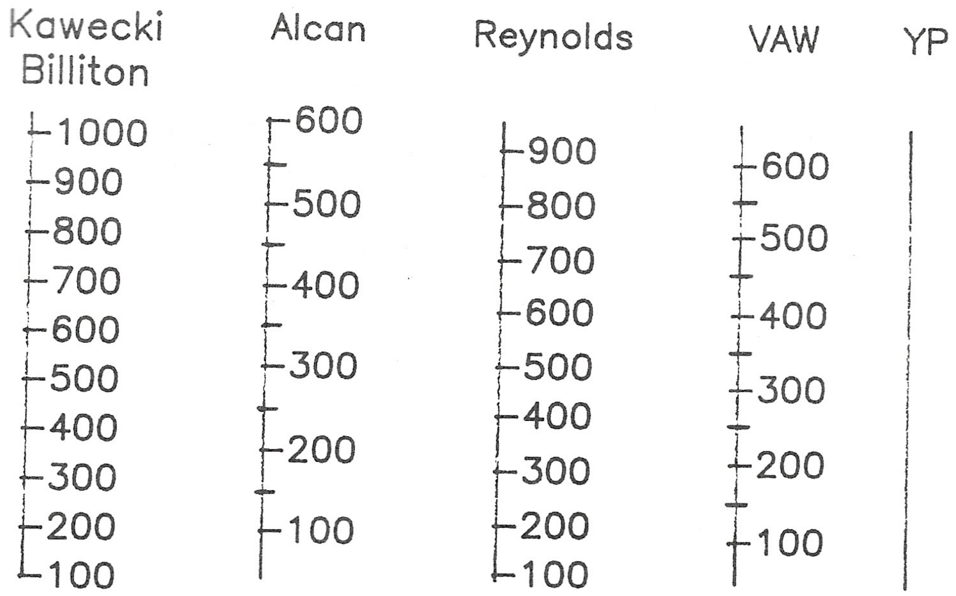
1. Kirby J.L., McCarthy R.W. and Levy S.A.
Proceedings of Light Metal 1986, pp. 749-757.
2. Maxwell I. and Hellawell A.
Metallurgical Transactions 1975, 23, pp. 229, 895 and 901

APPENDIX

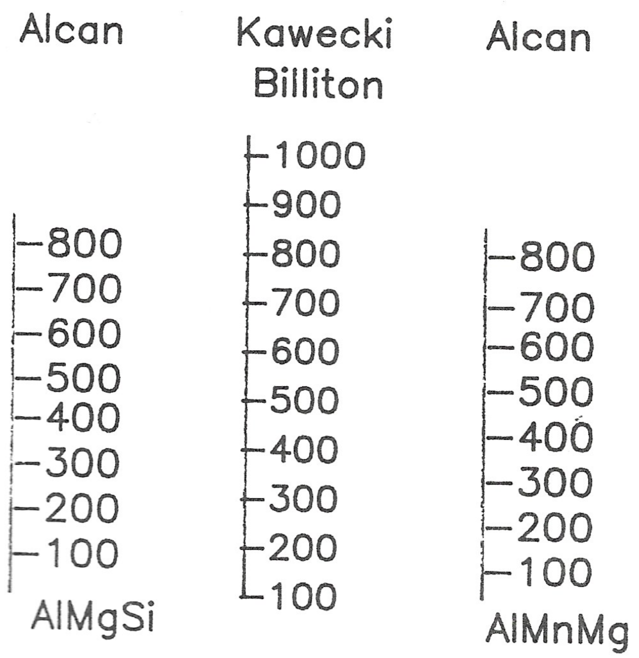
KAWECKI-BILLITON GRAIN REFINEMENT TEST

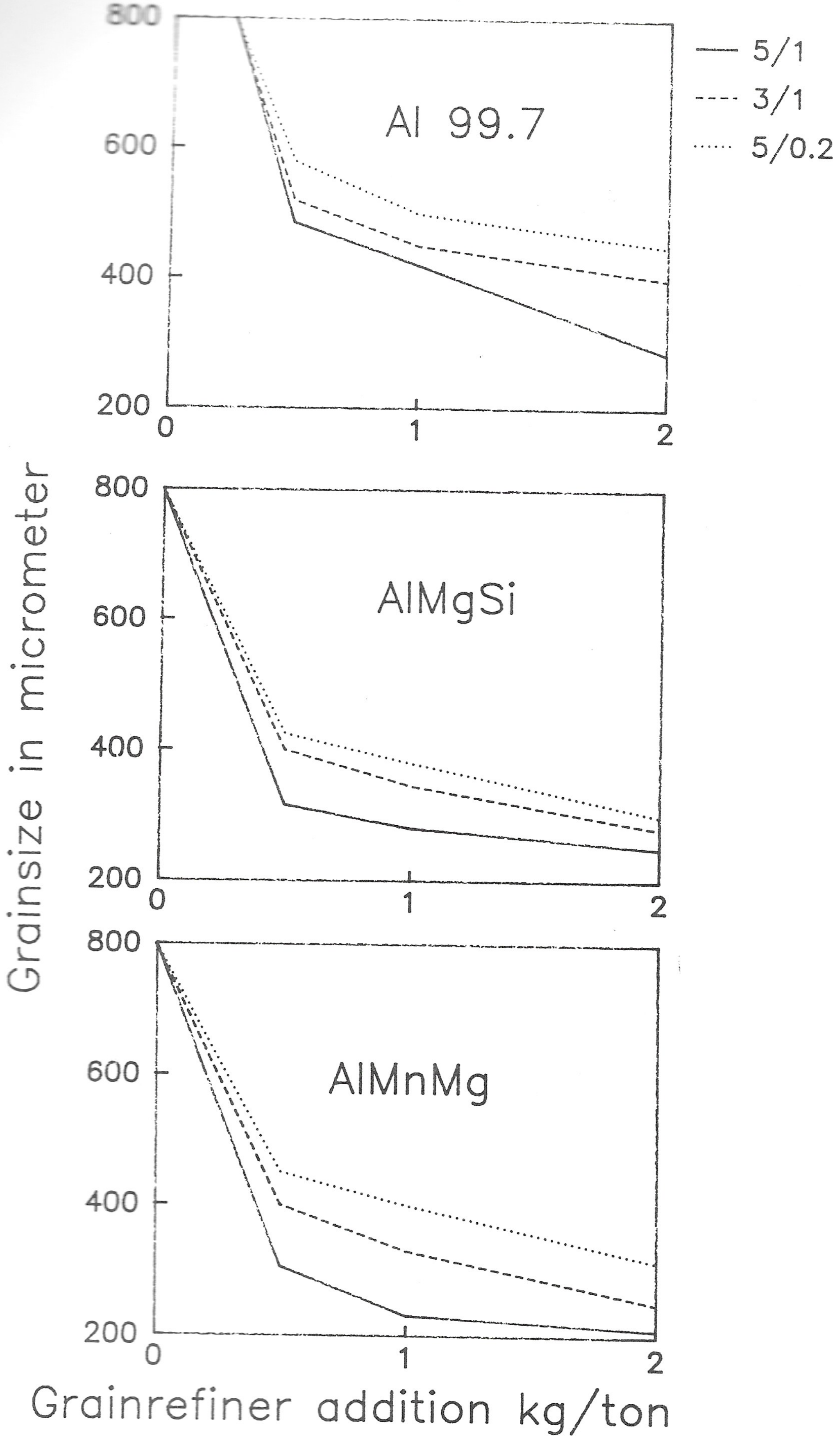
The Kawecki-Billiton grain refinement test, or ring test, is carried out according to the following procedure:
2 kg. of 99.7% virgin pure Aluminium is added to a crucible and held at a constant temperature of 720 ± 5 °C. during the test.
The addition of grain refiner rod to be tested is added, stirred into the melt for 15 seconds with a graphite rod and held for two minutes.
The treated Aluminium is poured into a ring which is placed on a refractory block (glassrock material).
Ring specification: 75 mm. in diameter, 20-30 mm. in height
5-7 mm. wall thickness, coated with graphite
The lower surface of the sample is etched (60% HCL, 30% HNO_3, 5% HF, 5% H_2O). The grains are counted by means of the intercept method, utilizing a stereo microscope with a 20x magnification.

Correlation for Al 99.7%

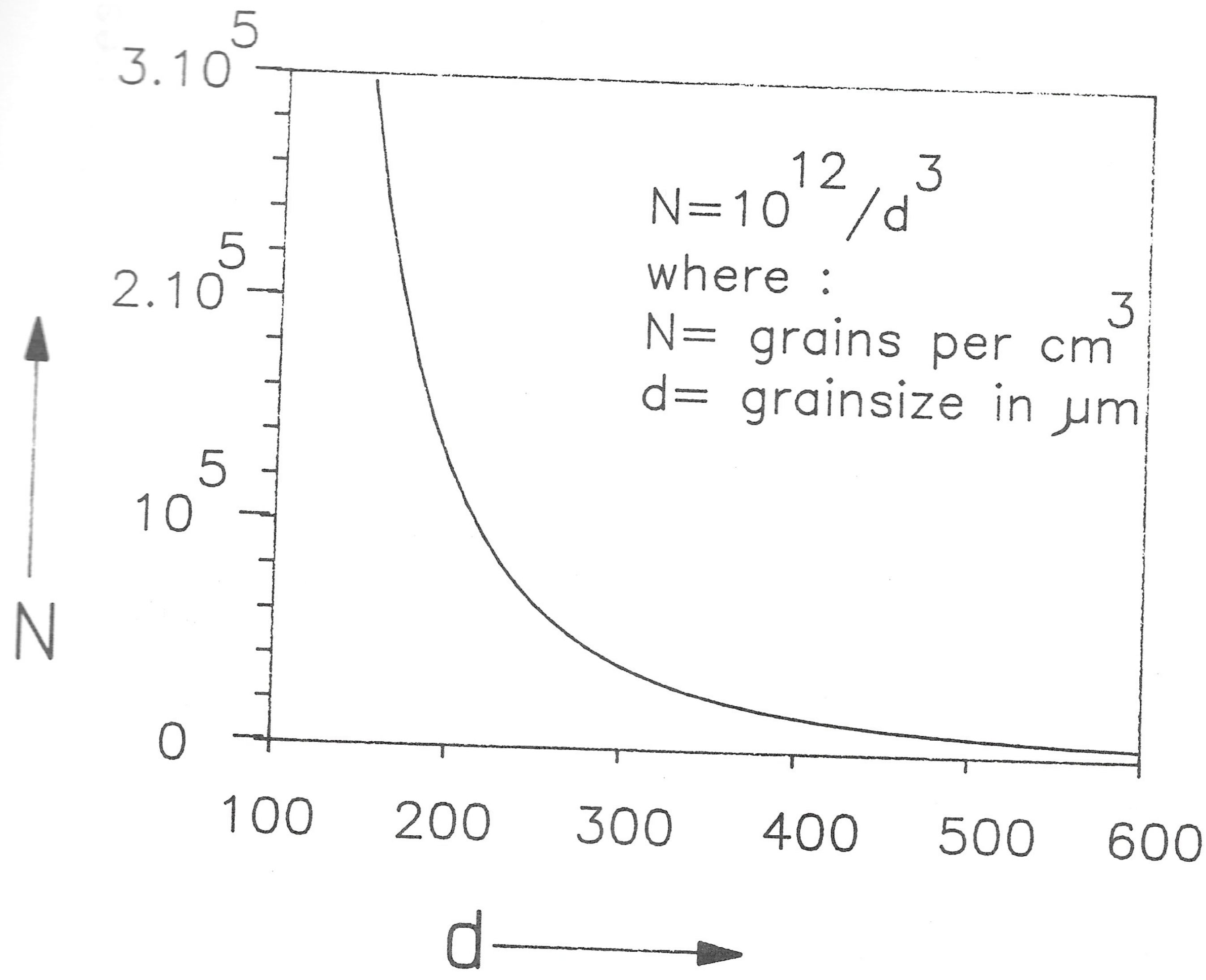


	Kawecki Billiton	Alcan	Reynolds	VAW	YP
Cooling Rate °C./s	0.2	5	0.8	5.5	?
Testing Time, min.	15	60	25	60	?





Al 99.7
AlMgSi
AlMnMg
5/1
3/1
5/0.2
800
600
400
200
0
1
2
Grainsize in micrometer
Grainrefiner addition kg/ton


3.10^5
2.10^5
10^5
0
N
N=10^12/d^3
where :
N= grains per cm^3
d= grainsize in µm
100
200
300
400
500
600
d

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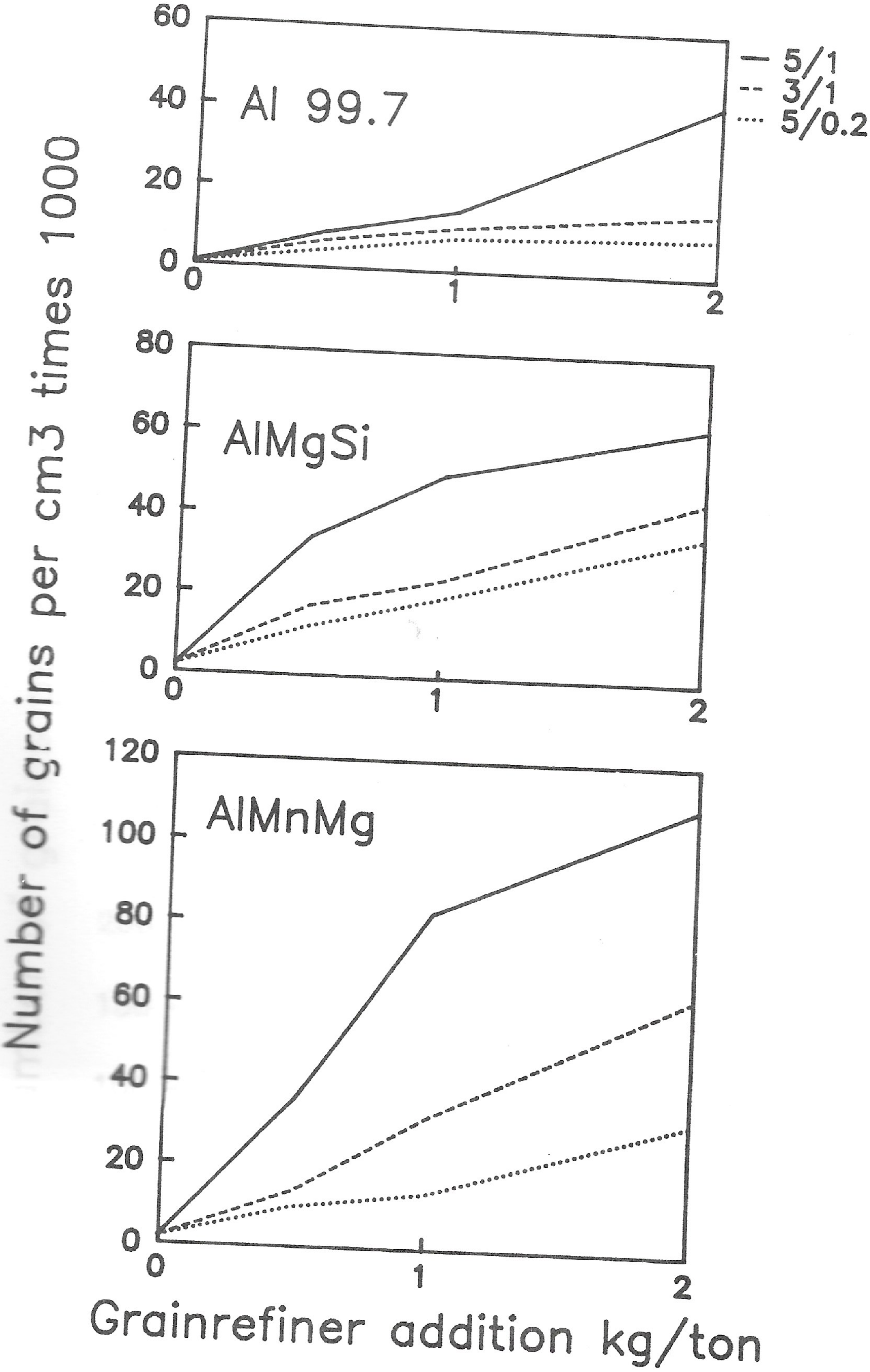

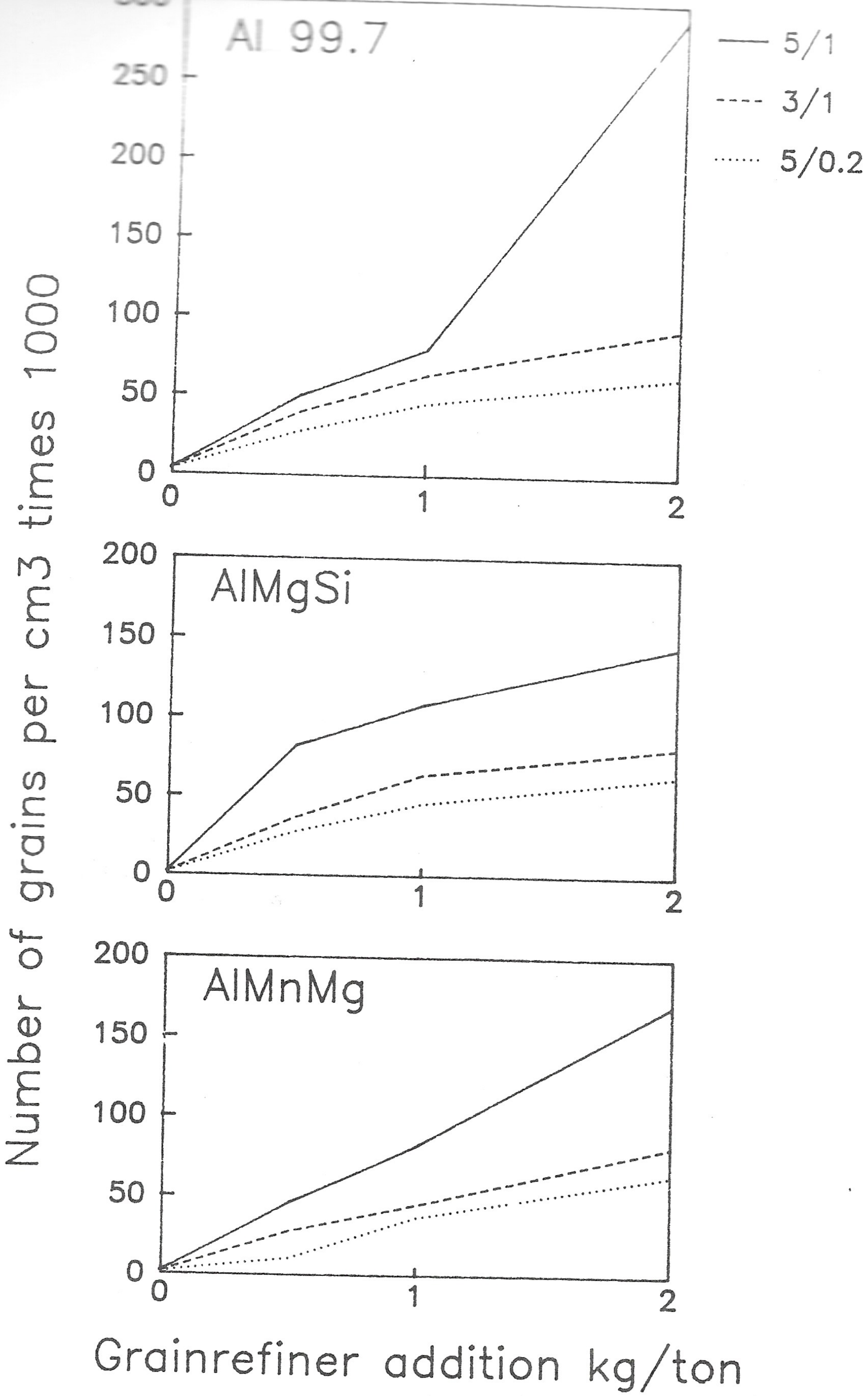
Al 99.7
250
200
150
100
50
0
0
1
2
— 5/1
---- 3/1
...... 5/0.2
Number of grains per cm3 times 1000
200
AlMgSi
150
100
50
0
0
1
2
200
AlMnMg
150
100
50
0
0
1
2
Grainrefiner addition kg/ton

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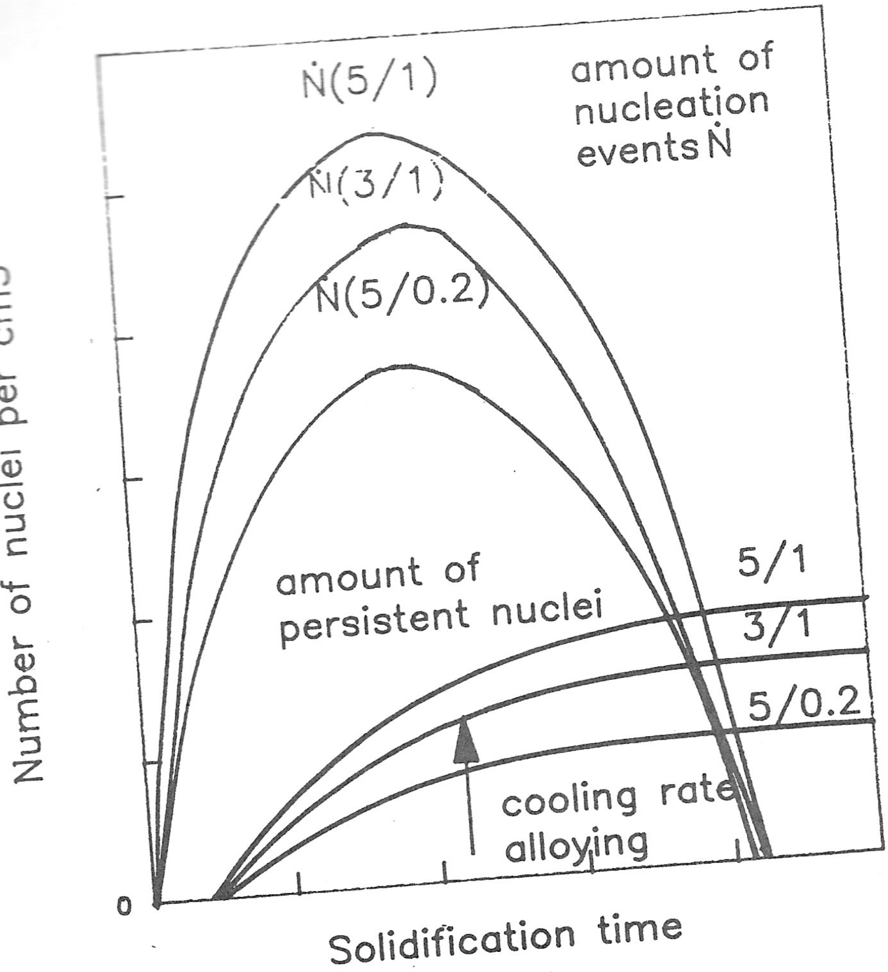
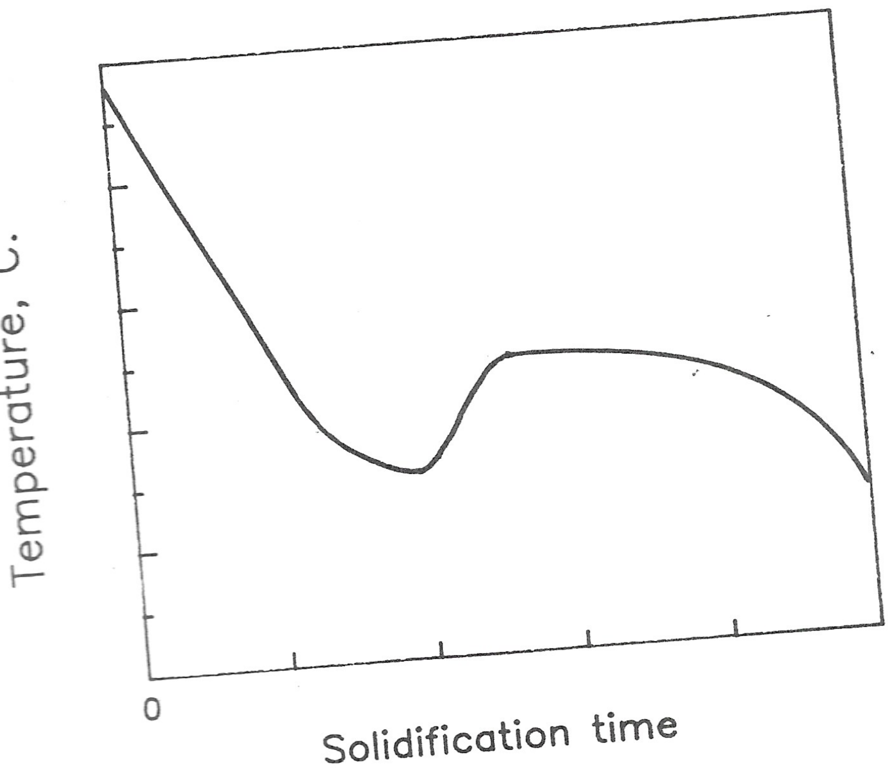


Ṅ(5/1)
amount of nucleation events Ṅ
Ṅ(3/1)
Ṅ(5/0.2)
Number of nuclei per cm3
amount of persistent nuclei
5/1
3/1
5/0.2
cooling rate alloying
0
Solidification time



Temperature, °C.
0
Solidification time