Novel Image Analysis to Determine the Si Modification for Hypoeutectic and Hypereutectic Al-Si Alloys

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The aim of this research is to develop a novel analytical approach to determine the level of modification of silicon in Al-Si hypoeutectic and hypereutectic alloys. Two standards to determine the level of modification of silicon particles were investigated: the AFS Si Modification Standard and the Primary Si Grade.[1,2] The first method can only be used for Al-Si hypoeutectic alloys and the second one for Al-Si hypereutectic alloys. In both methods, the determination of the level of modification of silicon is carried out by comparing micrographs of the test sample with the respective standards by simple observation. This results in a method with a bias error and does not take advantage of the analytical accuracy made possible by modern image analysis techniques. This paper presents a method known as image-analysis-based silicon modification level, which provides highly reliable results to determine the level of silicon modification for Al-Si hypoeutectic and hypereutectic alloys.

INTRODUCTION

Silicon modifiers are widely used in the Al-Si casting industry because they result in significant improvements to yield and tensile strengths, wear resistance, and elongation.[3,4] For Al-Si hypoeutectic alloys, the alkali elements (including strontium and sodium) are commonly used as silicon modifiers.[5] In the case of Al-Si hypereutectic alloys, phosphorous is usually added in the form of Al-P, Al-Cu-P, or Cu-8 wt.% P Al-Fe-P master alloys.[6–8] For instance, additions of phosphorous transform the primary silicon from star shaped into polyhedral and increase the number of primary silicon particles up to three times. In addition, the distance between particles is reduced ~50%.[7] Therefore, the precise determination of the level of modification of the microstructure is an effective and simple procedure to estimate the mechanical properties of Al-Si alloys.

In the literature, only two methods are found to characterize silicon particles in Al-Si alloys, one for hypoeutectic (American Foundry Society [AFS] Si Modification Standard) and the other for hypereutectic alloys (Primary Si Grade).[1,2,9] Djurdjevic et al.[1] reported successful results using the AFS Si Modification Standard and thermal analysis for on-line determination of the level of microstructure modification. Djurdjevic et al. developed a computer program capable of determining the AFS Si Modification Standard using image analysis. However, this method is limited to Al-Si hypoeutectic alloys. The main two limitations of the Primary Si Grade are that it is only applicable to Al-Si hypereutectic alloys and it only considers the diameter of the primary silicon particles, ignoring the Al-Si eutectic particles, which are always present in Al-Si hypereutectic alloys.[2]

Full refinement of the primary silicon particles is possible using current technology, transforming the microstructure of the Al-Si hypereutectic alloys into the appearance of a refined eutectic or hypoeutectic.[2] In order to compare the size of the silicon particles for different alloy compositions, a new standard is required. This research presents an unbiased characterization algorithm for Al-Si hypoeutectic and hypereutectic alloys for the analysis of the silicon particles in Al-Si alloys. This algorithm, image-analysis-based silicon modification level (SiML), is capable of evaluating primary and eutectic silicon particles separately or together. It is applicable for a large size distribution of silicon particles within a single analytical field, as shown in Figure 1. See the sidebar for experimental procedures to determine the key stereological characteristics of the silicon particles.

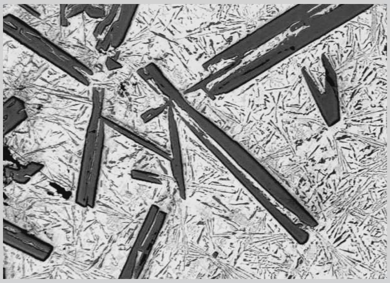

Figure 1. Micrographs showing the differences in size of Al-Si particles found in Al-Si hypereutectic alloys for distinct Si contents and magnifications: (a) 390.1(I) alloy at 500× magnification and (b) 393.2(II) alloy at 50× magnification.[2] (Figure 1a is a micrograph of a test sample cast with electromagnetically stirred and vibrated melt. The particular conditions and melt treatment methodology can be consulted in Reference 2.)

ANALYSIS OF THE RESULTS AND SiML METHODOLOGY

The statistical analysis to assess the SiML revealed that area and aspect ratio of the silicon particles could be used as main stereological parameters to distinguish the Al-Si eutectic from the primary silicon particles. Figure 2 depicts the limits in area and aspect ratio of the two silicon phases.

Figure 2 shows that the average area of the silicon particles has an overlap between the Al-Si eutectic and primary silicon particles. Only Al-Si eutectic particles exist between 0 μm^2 and 50 μm^2 in area and only primary silicon particles were observed larger than 1,100 μm^2. Moreover, 99.95% of the primary silicon particles have an area of less than 500 μm^2. Therefore, this was used as an upper limit for the Al-Si eutectic particles and a lower limit for the primary silicon ones. It is important to mention that the limit was established based on an analysis conducted for different 390.1 and 393.2 Al-Si hypereutectic alloy compositions, as shown in Table A. The mentioned samples were solidified under natural heat exchange conditions and using electromagnetic stirring and vibration. The stereological characteristics described in the sidebar can be used so long as the Al-Si eutectic and primary silicon particles can be separated.

On the other hand, silicon particles with areas between 50 μm and 500 μm need further analysis to determine the stereological characteristics that permit their separation. For this reason and because it is well known that primary silicon particles are polyhedral while Al-Si eutectics are plate-like, their aspect ratio was used as a stereological feature for their separation. The aspect ratio of 99.5% of the Al-Si eutectic is 2.3 or larger for the area range of 50 μm to 500 μm (Figure 2). In addition, for the same area range, 99.9% of the primary silicon particles have an aspect ratio smaller than 2.3 (Figure 2).

Table I. Results of the SiML for the Al-Si 3XX.X Alloys Based on Statistical Analysis of the Si Stereological Characteristics

SiML	Area (μm^2)	Perimeter Results (μm)
1	880.5	130.8
2	440.3	115.9
3	251.4	101.1
4	170.3	86.1
5	126.0	71.2
6	98.4	56.3
7	79.9	41.4
8	66.7	26.5

Note: The data in this table correspond to the results calculated for the different SiML using Equations 1 and 2 (Figure 3) and the micrographs presented in Figure 4.[2]

Only 0.5% of the Al-Si eutectic particles and 0.1% of the primary silicon ones fell within the range of primary silicon particles. The statistical analysis showed that of every 2,000 silicon particles measured (eutectic and primary), only 2.5 silicon eutectic particles were misidentified as primary silicon ones when judged by aspect ratio. On the other hand, of every 200 primary silicon particles measured only one was misidentified as Al-Si eutectic, which is equivalent to approximately seven micrographs at 200× magnification for the 390.1 Al-Si hypereutectic alloy solidified under natural heat-exchange conditions. Thus, the error of this method was considered negligible and the number of particles that were not properly separated had insignificant influence on the final results.

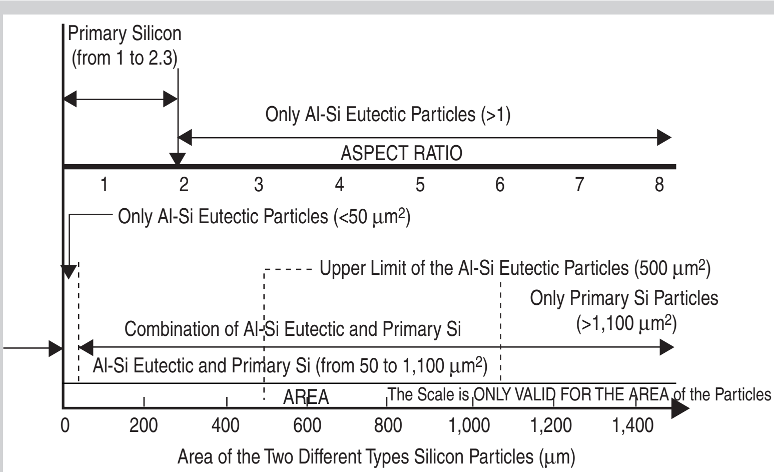

Figure 2. Stereological limits for the eutectic and primary silicon particles present in Al-Si alloys.

The analysis of the other measured stereological parameters is as follows: the number of silicon particles has no symmetry below SiML = 3. Length, width, or equivalent diameter displays an inflexion point at SiML = 5 then changes very little for small particles. This complicates the development of a standard scale to evaluate the level of microstructural modification of silicon particles for various compositions, or levels of refinement of the silicon particles based on the described stereological parameters (Figure A). Therefore, area and perimeter were selected to create the SiML standard because these two parameters present a continuous and monotonic decline with the SiML (Figure 3). Next, an analysis using linear and exponential regressions was conducted for perimeter and area, respectively (Figure 3).

From the general form of the relationship in Figure A, the perimeter was assigned a linear relationship plotted against the SiML. Figure 3 shows that the relationship chosen between the SiML versus area is exponential. For instance, by looking at the area of the silicon particles with an SiML less than

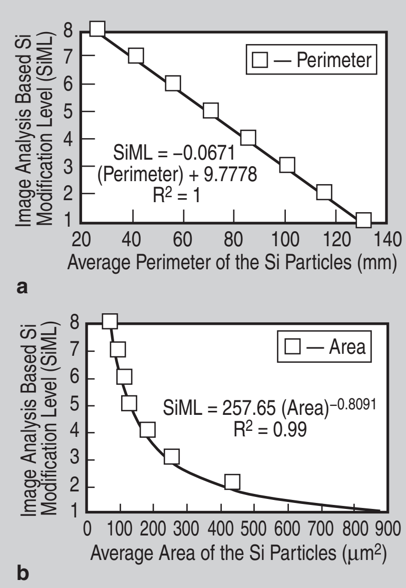

Figure 3. The results of the regression analysis of stereological parameters of silicon particles conducted for the assessment of the image-analysis-based silicon modification level (SiML) by using (a) perimeter and (b) area of the silicon particles. The linear behavior of the perimeter enables the assessment of the SiML.

EXPERIMENTAL PROCEDURE

The image-analysis-based silicon modification level (SiML) was developed by using the Leica Digital Model R and Q550IW image analysis system by taking 100 micrographs of various Al-Si hypoeutectic and hypereutectic compositions. The test samples used for this algorithm were solidified under natural heat-exchange conditions and using a novel electromagnetic stirring and vibration melt treatment.[2] Therefore, the silicon particles present in the different microstructures showed considerable differences due to the various levels of refinement of silicon. All test samples for the Al-Si chemical compositions (Table A) were prepared metallographically following the standard polishing procedures for observation using image analysis.

The micrographs were observed at various magnifications from 25× to 500×. Figure 1 shows light optical microscopy micrographs of the coarsest and finest silicon particles that were analyzed to develop the SiML methodology. The particles were measured using the image analysis system to assess the following particle characteristic stereological parameters: average area, average perimeter, average length, average width, average roundness, average aspect ratio, average equivalent diameter, and number of particles. The micrographs were ranked individually for each of the stereological parameters and divided into eight groups corresponding to eight silicon modification levels. Figure A shows the curves that relate the area, perimeter, and number of particles for the 100 different micrographs.

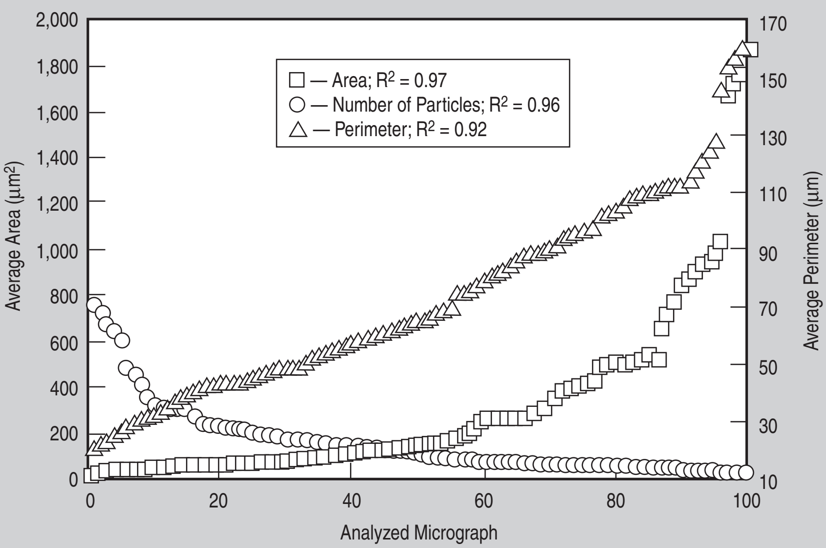

Figure A. The effect of the stereological parameters—average area, average perimeter, and number of particles—for the 100 analyzed micrographs.

Table A. Chemical Composition (in wt.%) of the Investigated Al-Si Hypo- and Hypereutectic Alloys Used to Cast the 100 Test Samples Analyzed to Determine the SiML Methodology[2] and the Respective Liquidus Temperature as Determined Using the Silicon Equivalent Method.[10]

AAA Designation	Si	Cu	Fe	Mg	Mn	Ni	T_{LIQ} (°C) Calculated
C355	4.85	1.03	0.09	0.14	0.01	0.05	626.8
308.0	4.89	3.85	0.09	0.16	0.01	0.02	622.3
328.0	7.00	0.96	0.29	0.21	0.01	0.01	611.3
W319	7.18	4.68	0.17	0.26	0.01	0.03	605.6
322.2(I)	9.12	1.18	0.18	0.28	0.01	0.05	596.7
322.2(II)	9.85	4.38	0.14	0.27	0.01	0.07	587.1
336.2	10.84	0.94	0.11	0.19	0.01	0.06	584.6
332.2	10.55	4.36	0.13	0.17	0.01	0.08	568.0
390.1(I)	13.14	4.11	0.51	0.99	0.21	2.19	600.2
390.1(II)	15.53	3.20	0.62	0.62	0.29	0.62	634.6
393.2(I)	25.0	5.64	0.64	0.09	0.15	0.1	756.9
393.2(I)	25.0	1.18	0.39	0.05	0.12	0.4	752.5
393.2(II)	24.16	2.46	0.93	0.17	0.36	1.25	738.4
393.2(III)	28.64	2.43	0.90	0.15	0.36	1.32	794.9

4, a rapid change can be observed. For an SiML greater than 4, however, the response approaches asymptotic, so it does not adequately separate the silicon particles. In order to avoid this limitation using area as an analytical parameter, it is recommended to use the perimeter to determine the SiML. In fact, a linear behavior of the stereological parameter would be the ideal case, since it attains the same level of confidence through the entire range of SiML.

The Primary Si Grade scale is a function of the diameter of the primary silicon particles. Because the precise determination of the equivalent diameter of elongated silicon particles is impractical, serious limitations of using the diameter of the silicon particles as an analytical parameter exist (Figures 1 and A). Image analysis enables the perimeter to be easily used as a parameter for determining SiML with the previously discussed advantages.

Based on the results of Figure 3, the regression equations for perimeter and area are:

$$\text{perimeter} = -14.904\ (\text{SiML}) + 145.73 \qquad R^2 = 0.99 \quad (1)$$

$$\text{area} = 1{,}112.3\ (\text{SiML})^{-1.3536} \qquad R^2 = 0.97 \quad (2)$$

Using Equations 1 and 2, the area and perimeter of the silicon particles present in the microstructure of an Al-Si alloy test sample can be determined if the SiML is known. It is also possible to determine the SiML based on the average area and perimeter of the silicon particles with the equations shown in Figure 3a and b.

A numerical regression of the data obtained from image analysis for the 100 micrographs (Figure A) was conducted to determine the SiMLs. The aim was to obtain Figure 3a and b and Equations 3 and 4 for area and perimeter, respectively. The corresponding correlation factors for Equations 3 and 4 are $R^2 = 1$ and $R^2 = 0.99$, correspondingly, and are the proof of the high accuracy level of the SiML. The numerical values as determined using Equations 3 and 4 are presented in Table I showing the corresponding value for the different SiML levels. Based on the values determined using Equations 3 and 4 for the different SiMLs, eight micrographs were found to correspond to the closest value of perimeter to the

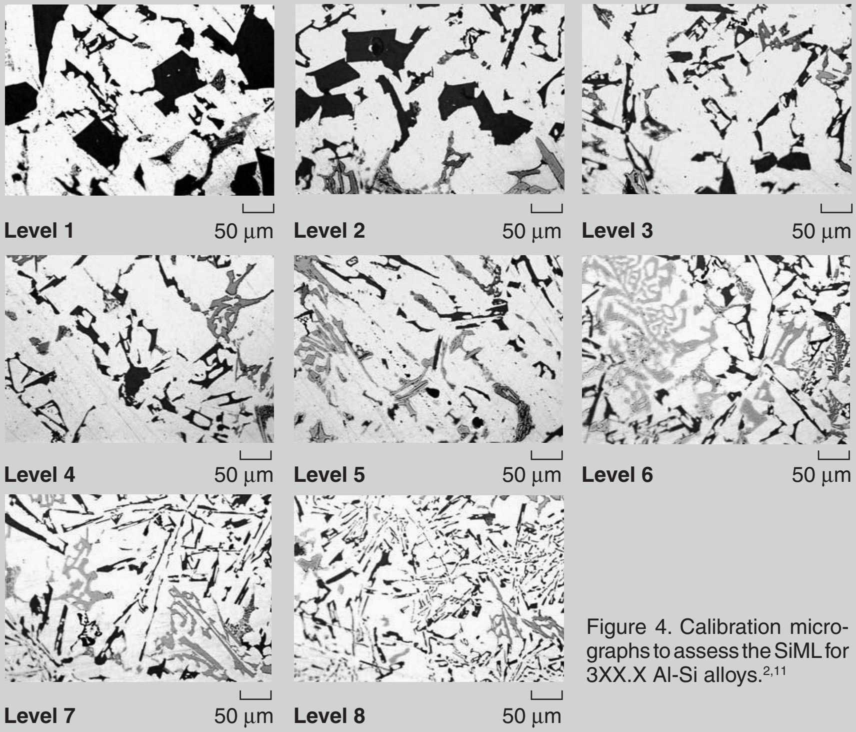

Figure 4. Calibration micrographs to assess the SiML for 3XX.X Al-Si alloys.[2,11]

one determined using Equation 3 based on the micrographs analyzed. The corresponding micrographs for the SiML determination using Equation 4 (for area) are not presented in this paper because perimeter attained the highest level of confidence since it is represented by a linear regression equation. Figure 4 shows the eight micrographs for the respective SiML levels that were used to develop the standard.

$$\text{SiML} = -0.671\ (\text{perimeter}) + 97{,}778 \qquad R^2 = 1 \qquad (3)$$

$$\text{SiML} = 257.65\ (\text{area}) \qquad R^2 = 0.99 \qquad (4)$$

Equations 3 and 4, which determine the SiML as a function of perimeter and area (Figure 3), were used to create a computer program that determines the SiML based on the image analysis results. Therefore, SiML is determined automatically as the image analysis is conducted. It is also important to mention that the SiML was designed to be determined in three different ways: using Equations 1 and 2, comparing the results of image analysis with the results presented in Table I, and matching micrographs of the analyzed test samples with the ones presented in Figure 4. Furthermore, the assessment of the SiML can also be carried out by calculating the area fraction of the silicon particles with a lower or higher aspect ratio than 2.3. In order to determine the SiML by comparison using the micrographs presented in Figure 4, it is important to print analytical micrographs at 200× magnification and at the same size of the standard.

EVALUATION OF THE 390.1(I) Al-Si HYPEREUTECTIC ALLOY

Six die-cast pistons and six optical-emission spectroscopy (OES) test samples were used to assess the SiML and the Primary Si Grade method. Six samples were extracted from pistons cast using electromagnetically stirred and vibrated melt. The overall idea of this exercise was to present the SiML as a more reliable method than Primary Si Grade. It is important to mention that both the pistons and OES test samples were chemically modified using 60 ppm of phosphorous as a primary silicon modifier in the form of Cu-8 wt.% P master alloy. Figure 5 shows the microstructure of a die-cast piston. The summary of the results of the Primary Si Grade and the SiML for all the test samples are presented in Table II. See Reference 2 for more information about the conditions of the electromagnetic stirring and vibration melt treatment(s) for the alloys.

The data from Table II was plotted in Figure 6 to compare the spatial resolution of the SiML technique versus the Primary Si Grade method. As can be observed, the SiML is capable of distinguishing small differences in the primary silicon particles while the Primary Si Grade is a very rough estimation.

RESULTS AND CONCLUSIONS

The accuracy of the presented method is higher than the two standards previously reported since these standards were developed to obtain the level of silicon modification by simple observation of the micrographs against the sample.

The methodology developed by Djurdjevic et al.[1] is image-analysis based. Its main limitation is that it can only be used for analysis of the respective micrographs of the AFS Si Modification Standard for the different grades. This means that only one micrograph per grade was analyzed, in contrast with the SiML method in which 12.5 micrographs of various Al-Si alloys were used per SiML. Additionally, the AFS Si Modification Standard does not provide a full stereological analysis to determine the AFS Si Modification

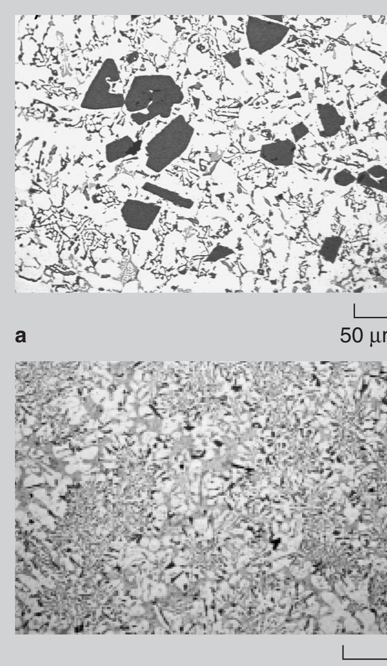

Figure 5. A representative micrograph of the microstructure of die-cast 390.1 alloy pistons (a) regular production ($T_{Pouring}$ = 750 ± 10°C and (b) electromagnetically stirred and vibrated treated melt ($T_{Pouring}$ = 602°C). The cooling rate of both pistons was similar (~1.6°C/s), the micrographs were taken at 200× magnification.[2,11]

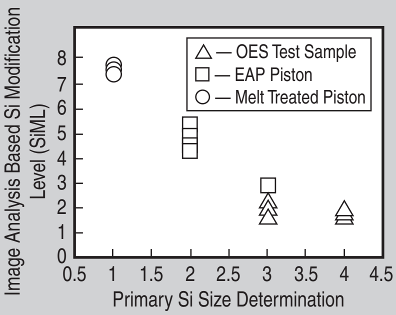

Figure 6. A comparison of the results of determining the Primary Si Grade and SiML for test samples extracted from the die-cast regular production, electromagnetically stirred and vibrated, and pistons and OES samples.[2] All samples were cast with 390.1(I) alloy.

Standard based on the results of image analysis. Furthermore, this method is limited only for Al-Si hypoeutectic alloys.

The use of diameter (in this case equivalent diameter) to determine the level of modification of silicon particles is inaccurate, therefore the Primary Si Grade is a biased method. Additionally,

Table II. Image Analysis Results and Comparison between the Primary Si Grade and the SiML for Die-Cast Pistons using Untreated and Electromagnetically Stirred and Vibrated Melt*

Test Sample Type	Average Area of Primary Si Particles (μm^2)	Average Perimeter of Primary Si Particles (μm)	Primary Si Size Det.	SiML
OES	498.2 ± 35.4	27.4 ± 7.1	3	1.89
	314.1 ± 40.6	30.1 ± 2.5	3	2.20
	465.2 ± 50.6	29.4 ± 9.8	3	2.05
	524.6 ± 65.8	33.6 ± 23.4	4	1.96
	568.9 ± 35.1	35.9 ± 17	4	1.84
	607.7 ± 50.0	38.5 ± 15	4	1.75
Regular Production Pistons	143.7 ± 28.5	17.8 ± 4	2	4.80
	142.7 ± 28.2	17.6 ± 6.6	2	4.81
	178.4 ± 25.9	20.1 ± 4.5	2	4.24
	172.1 ± 34.0	18.9 ± 2.8	2	4.35
	124.5 ±42.4	16.7 ± 3.1	2	5.25
	269.0 ± 45.1	23.5 ± 7.1	3	2.80
Laboratory Die-Cast Pistons (Electromag. Stirred and Vibrated Melt)	72.0 ± 12.3	5.3 ± 1.2	1	7.56
	70.7 ± 14.6	3.7 ± 0.8	1	7.66
	73.4 ± 8.2	7.3 ± 1.1	1	7.45
	71.6 ± 5.9	8.6 ± 2.3	1	7.59
	69.8 ± 4.6	4.2 ± 0.7	1	7.73
	72.9 ± 15.6	5.2 ± 1.6	1	7.49

*Both pistons were cast using 390.1 Al-Si hypereutectic alloy.[2,10]

this method is limited to the analysis of primary silicon particles and to Al-Si hypereutectic compositions. Figure 6 and Table II show the significantly higher accuracy of the SiML in determining the level of modification of silicon particles. Figure 6 also shows that for highly refined microstructures (i.e., test samples cast using melt treated with the electromagnetic stirring and vibration methodology) the Primary Si Grade does not have the required resolution to determine the level of modification of silicon particles.

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