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Statistical Analysis of Structure Loss in Czochralski Silicon Growth

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Abstract. In Czochralski monocrystalline silicon growth, structure loss (SL) is the loss of the mono-crystalline structure. It represents a significant loss of productivity. In this work, this phenomenon is investigated by statistical analysis of production data of roughly 14000 ingots produced over a year of time at NorSun factory in Årdal, Norway. It is found that ingots with structure loss typically have lower heater power and temperature fluctuations than ingots without structure loss after four hours of body (ca. 240 mm). Particularly, ingots without manual adjustment by furnace operator have significantly higher frequency of structure loss than ingots for which the operator has increased the temperature one or more times. Most ingots with structure loss are also found to have a higher pull speed on average than ingots without structure loss, and that there is a threshold below which no ingots had structure loss. A binary logistic regression was used for classification of ingots with and without structure loss and 30% of the data was used to comparison of predictions of the model. Using only the standard deviation of the temperature fluctuations around a moving average provided a prediction accuracy of 99.6%, for ingots that have passed six hours of body (ca. 360 mm).

INTRODUCTION

The Czochralski process is the main method for producing monocrystalline silicon for high-efficiency solar cells. Improving the yield and reducing production time contribute to continuing the cost reduction of this process. The yield and production time are commonly impaired by transition from monocrystalline to multicrystalline structure, called structure loss. It is typically preceded by slip and dislocation formation up to one ingot diameter from the solidification front and up into the already formed crystal, and this region is also out of specification.

Possible causes of structure loss include high thermal stress, fluctuation of the crystallization rate with temporal remelting due to turbulent melt flow, "W-shape" of the solidification front, particles contacting the solidification front, and mechanical vibration of the crystal or waves on the melt [1]. Stockmeier [2] also considered the amplitude and frequency of temperature fluctuations crucial for dislocation formation as well as the length of the edge facets. He concluded that dislocations most likely originated near one of the growth ridges. Growth ridges are protrusions from cylindrical cross-section due to faceted growth, forming four vertical ridges on ingots grown in the [100] direction.

The present work comprises statistical analysis of industrial data in relation to structure loss. The objective is to test hypotheses for causes of structure loss and determine which parameters that are most important for controlling it. Access to a large dataset allows for inferences and statistical learning.

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METHODOLOGY

Three statistical methods are used to analyze industrial process parameters, namely hypothesis testing, relative importance of parameters by feature selection and prediction of structure loss based on a binary logistic regression, and they are described in following subsections. The data treatment procedure is visualized in Fig. 1.

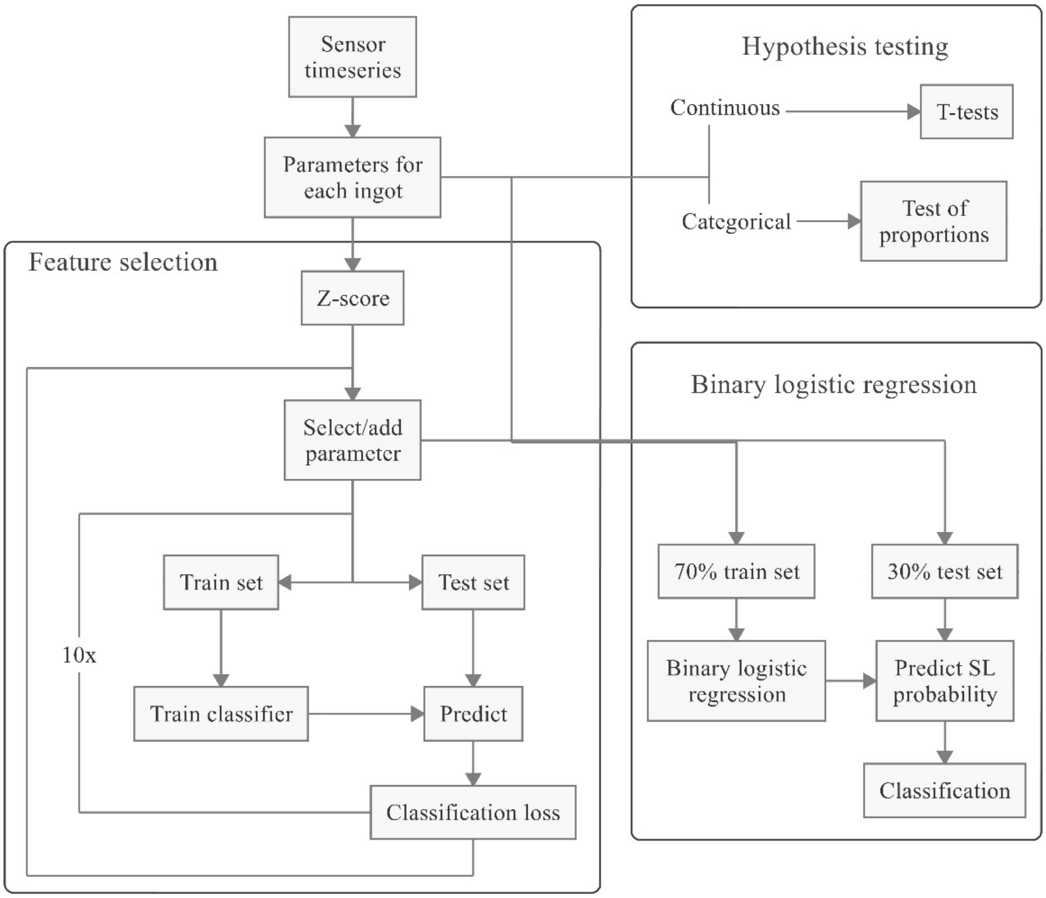


FIGURE 1. Schematic view of the treatment of process data.

The industrial data is collected over approximately one year and includes 13929 n-type ingots. In order to assess the parameters at the time of structure loss, calculations are sometimes based on the last hour of growth of structure loss ingots, and not the entire body as for ingots without structure loss. Fluctuations are for some parameters determined from a moving average, for which the window size is selected from visual inspection in order to smooth rapid fluctuations while still tracking the trend of the signal. The following parameters are computed for each ingot from stored continuous sensor readings:

- Standard deviation of heater power fluctuations, calculated as the difference between the signal and a moving average, for the entire body of ingots without structure loss and the period 110-50 min before end of growth for ingots with structure loss, which is the last hour of the moving average. The moving average is calculated for a window size of 75 min.
- Standard deviation of temperature fluctuations, calculated as the difference between the signal and a moving average, for the entire body of ingots without structure loss and the period 85-25 min before end of growth for ingots with structure loss, which is the last hour of the moving average. The moving average is calculated for a window size of 37.5 min.
- Number of heater power spikes after four hours of body. The difference between two adjacent heater power readings, with 30 s time interval, are calculated and spikes are counted where the difference exceeds 6 and 10 standard deviations of all differences between adjacent readings.

- Pull speed average in region with fluctuation around a typically constant pull speed, excluding first six hours of body and the end of full-length ingots. For ingots with structure loss that passes six hours of body and ends in the region with fluctuations around a typically constant value, the average is calculated for the last hour of the ingot.
- Standard deviation of pull speed fluctuations, calculated as the difference between the signal and a moving average, excluding the first six hours of body and the end of full-length ingots. For ingots with structure loss that passes six hours of body and ends in the region with fluctuations around a typically constant pull speed, the standard deviation of the pull speed is calculated for the last hour of the ingot.

The data also includes categorical factors, including type of production (customer), three different crucible types (Crucible 1-3), whether cooling jacket is used or not, and tree different feedstock mixtures used for re-charging. The cooling jacket enhances the cooling rate of the crystal above the melt. The analyses typically differentiate between A- and B-ingots, where an A-ingot is the first ingot of a run and the B-ingot is the second ingot, after recharging the crucible in the furnace.

Hypothesis Testing

The above parameters are compared for ingots with and without structure loss by two-sample t-tests of ingots with and without structure loss for continuous parameters and by test of proportions of structure loss ingots for categorical parameters. The data is typically filtered to only represent the most frequent type of production, Crucible 1, cooling jacket not used and Feedstock 1 used after A-ingots. The following hypotheses are tested:

1. Heater power and temperature fluctuations: Increased fluctuations can increase the frequency of local and temporal remelting of the solidification front, and thus increase the frequency of structure loss.
2. Heater power spikes: Manual adjustments of temperature by operators prevent cold melt and ingots growing out of shape with increased risk of structure loss. If there are no heater power spikes due to manual adjustments, the chance of structure loss may be increased.
3. Pull speed: If ingots with structure loss have reduced pull speed average and increased fluctuations, it may be explained by increased frequency of local and temporal remelting of the solidification front. On the other hand, if ingots with structure loss have increased pull speed average, the cause might be the increased interface deflection and increased thermal stresses near the solidification front [1].
4. Diameter: Larger diameter increases thermal stresses, which may explain a higher proportion of ingots with structure loss grown with larger diameters.
5. Cooling jacket: Increased thermal gradient and thermal stresses in ingots pulled with cooling jacket, may explain an increase in structure loss frequency with cooling jacket.
6. Crucible: Different crucibles may release particles at different rates, for instance when bubbles rupture as the inner crucible wall dissolves [3], affecting the frequency of structure loss.
7. Feedstock: Different feedstocks can potentially cause different structure loss frequency, for instance by presence of impurities that can form particles that can cause structure loss when the particles come in contact with the solidification front.

Relative Importance of Parameters

The relative importance of the continuous parameters is found using Sequential Feature Selection. This method is based on workflow highlighted in Fig. 1 and it uses Machine Learning technique to test the importance of each parameters or the combination of them. The meaning of the most important parameters is here the parameters that provide best prediction of structure loss and are selected first by the feature selection algorithm. In order to avoid influence due to different order of magnitude of the parameters, each input parameter has been standardized by subtracting its mean and dividing it by its standard deviation to find the z-score. Two machine learning techniques have been implemented in a MATLAB® script, namely Support Vector Machine (SVM) and Gradient Boost Tree classifiers (GBT) [4]. 10-fold cross-validation is performed and the classification loss is summed for each prediction and divided by the number of test ingots. This mean value is used to evaluate different subsets of parameters. The parameter that gives the lowest mean value for the classification loss is first selected, and the next parameter selected gives the lowest loss in combination with the previous selected parameter. In this way, parameters are included sequentially.

Predictive Model

A binary logistic regression is used for classification, in which structure loss is predicted if the probability calculated by the regression is greater than 0.5. The data is randomly divided in 70% for making the regression and 30% for testing its predictive power. Different combinations of parameters were included in the binary logistic regression, in order to find the smallest number of parameters that gives high accuracy of prediction.

RESULTS AND DISCUSSION

The statistical analysis includes first hypothesis testing of mean values for continuous variables between ingots with structure loss and without structure loss, and comparison of the proportion of ingots with structure loss for different factors. Then, structure loss is predicted by classification based on a binary logistic regression, and the classification is presented for parameters which best predict whether an ingot has structure loss.

Hypothesis Testing

A t-test finds that the mean of power fluctuations around a moving average is significantly higher in the last 110-50 min of ingots with structure loss than for the body of ingots without structure loss. The same observation is made for both A-ingots and B-ingots. This is however most likely a symptom of structure loss and not a cause, because ingots with structure loss are typically short and the power fluctuates the most in the beginning of the ingot. Thus, the analysis is restricted to ingots lasting long enough for the fluctuations to stabilize, namely four hours into the body (ca. 240 mm).

After four hours of body, the standard deviations of both heater power and temperature fluctuations are significantly lower for ingots with structure loss than ingots without structure loss (Fig. 2(a) and (b)), both for A- and B-ingots. Thus, the tests do not support the hypothesis that increased heater power and temperature fluctuations may increase the frequency of structure loss due to increased frequency of local and temporal remelting at the solidification front. In the case of ingots longer than four hours of body, the fluctuations in heater power and temperature is in a large part due to spikes in the heater power following manual adjustments by operators. Such adjustments are made to increase the temperature of the melt and prevent the ingot from growing out of shape and reduce the risk of structure loss. It is shown in Fig. 2(c) that the proportion of ingots with structure loss after four hours of body is significantly higher if the temperature is not adjusted so that adjustments of the temperature successfully reduces the risk of structure loss. It may also suggest that a too cold melt, assumed more frequent for ingots without temperature adjustments, is an important cause of structure loss. The hypothesis that manual adjustments helps to prevent ingots growing out of shape and reduces the risk of structure loss is supported. It is optimal for fast pulling and high productivity to have a low temperature of the melt, but it can become too cold in part due to differences between furnaces which is why supervision is necessary. This analysis shows the importance of operator supervision and more careful supervision could potentially reduce the frequency of structure loss.

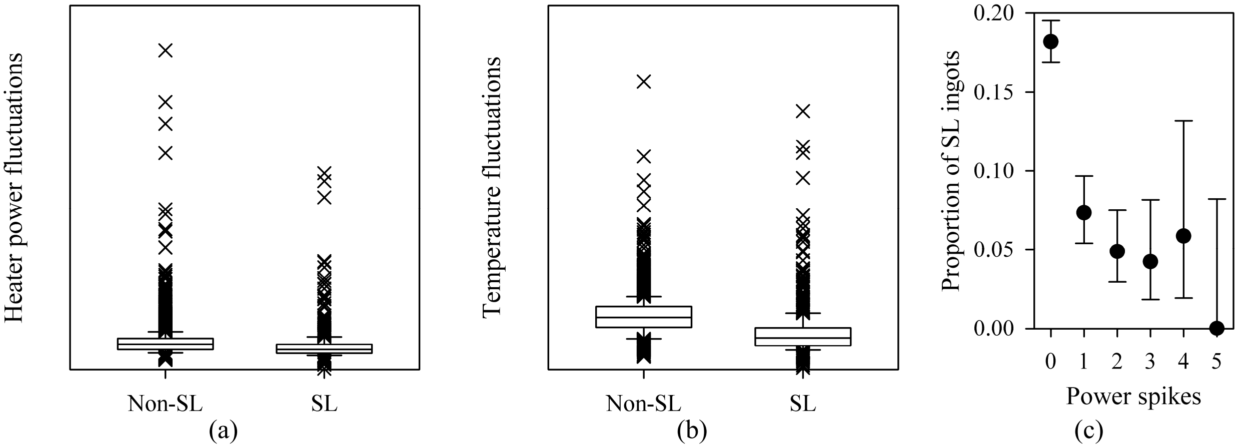


FIGURE 2. Box plot of standard deviation of fluctuation about moving average for heater power (a) and temperature (b) signals after four hours of body for ingots with structure loss (SL) and without structure loss (Non-SL), and proportion of ingots with structure loss after four hours of body as function of number of spikes in heater power signal (c).

Figure 3 shows box plots of pull speed average (a) and fluctuation (b) for ingots with and without structure loss. The mean of pull speeds for ingots with structure loss is slightly, but significantly higher in the last part of ingots with structure loss than for ingots without structure loss, although this is not the case for A-ingots. There is also a threshold for which there are no structure loss ingots with lower pull speeds. This is not unambiguous supports of the hypothesis that increased pull speed average increases the tendency for structure loss due to increased interface deflection and thermal stress near the solidification front. The pull speed fluctuation is significantly lower in the last part of ingots with structure loss than for ingots without structure loss, unless filtering only A-ingots. This does not support the hypothesis that increased pull speed fluctuation may indicate increased frequency of local and temporal remelting at the solidification front and cause more frequent structure loss.

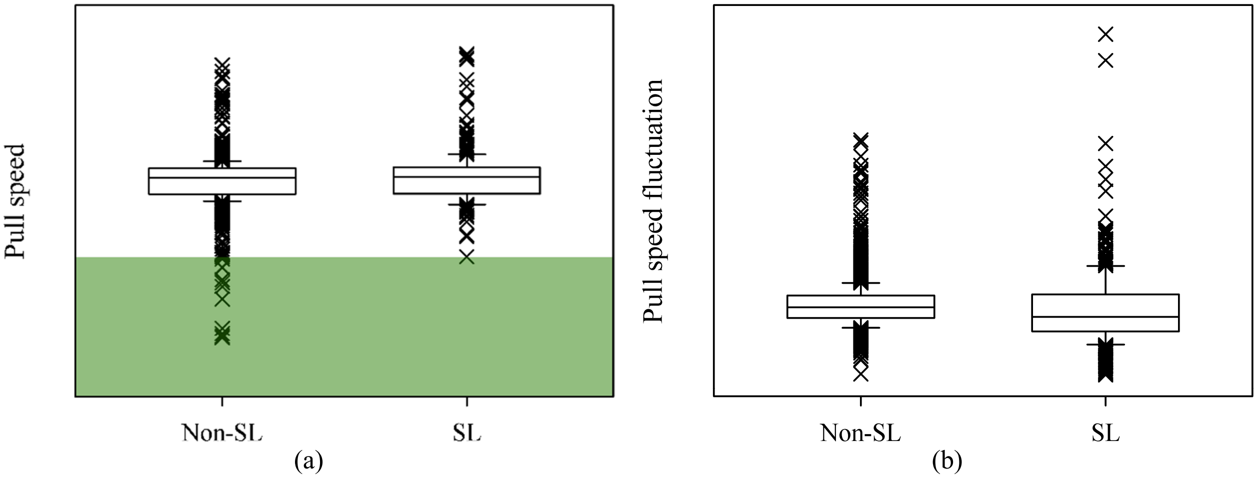


FIGURE 3. Box plot of pull speed average (a) and standard deviation (b) after 6 hours of body, where the signal fluctuates around a constant value, for the last hour of structure loss ingots (SL) and for the entire region of approximately constant value for ingots without structure loss (Non-SL). Only ingots without structure loss are found in the green region of subfigure (a) at relatively low pull speeds.

The effect of diameter on the frequency of structure loss is shown in Figure 4(a). 8.4 inch (213 mm) diameter gives a significantly higher proportion of ingots with structure loss than for 6.7 inch (170 mm) diameter ingots. This was however not the case for A-ingots. With the number of ingots in Table 1, the proportion of ingots with structure loss is not significantly higher for 8.4 inch diameter ingots compared to 6.5 inch (165 mm) ingots. It is thus not clear if increasing diameter gives more frequent structure loss due to increased thermal stress.

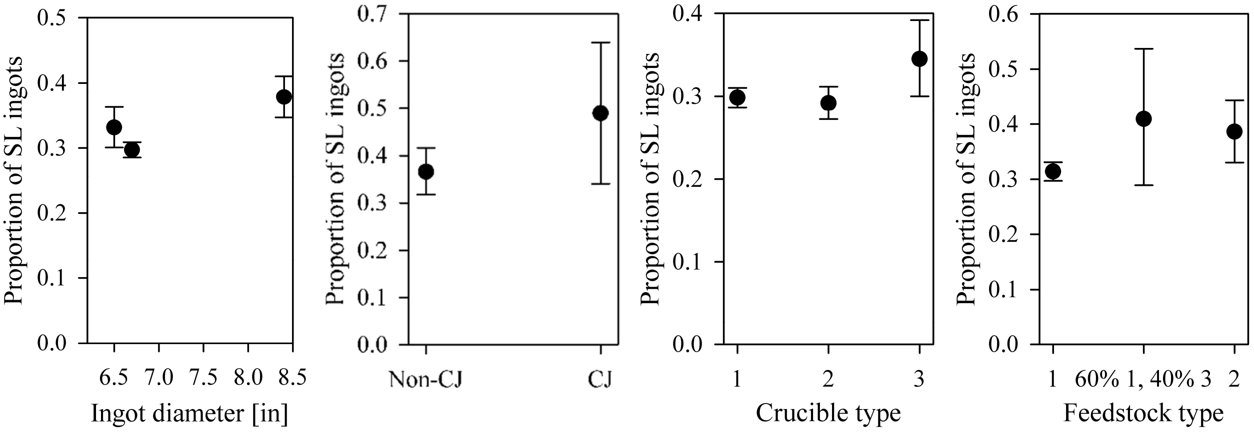
For ingots grown with cooling jacket, there is a higher proportion of ingots with structure loss in Fig. 4(b), but it is not significant with the number of ingots in Table 1. Thus it is not conclusive whether higher thermal gradients and thermal stress cause more frequent structure loss when using cooling jacket.

Use of different types of crucibles resulted in higher proportion of ingots with structure loss with Crucible 3 than Crucible types 1 and 2 for B-ingots, but not other ingots, as shown for all consecutive ingots in Fig. 4(c). There is thus not unambiguous evidence for different crucibles releasing particles at different rates.

There is a significant difference in the proportion of ingots with structure loss between Feedstock 1 and 2 in Fig. 4(d). Detailed information about the difference between the feedstocks and the cause of the difference in proportion of ingots with structure loss are not known yet.

TABLE 1. Number of ingots in the analysis of proportion of ingots with structure loss in Fig. 4.

Diameter	# ingots	Cooling jacket	# ingots	Crucibles	# ingots	Feedstock	# ingots
6.5 inch	911			Crucible 1	5809	Feedstock 1	3000
6.7 inch	6012	Non-CJ	380	Crucible 2	2071	60% 1, 40% 3	66
8.4 inch	928	CJ	47	Crucible 3	432	Feedstock 2	298



(a) (b) (c) (d)

FIGURE 4. Proportion of ingots with structure loss with 95% confidence interval for different ingot diameters (a), use of cooling jacket (CJ) (b), crucibles (c) and feedstock material (d). A-ingots are not included in part (d), and 60% 1, 40% 3 denote a mixture of 60% of Feedstock 1 and 40% of Feedstock 3.

Relative Importance of Parameters

Feature ranking was performed to identify the most important parameters. Forward Sequential Feature Selection with the Support Vector Machine and the Gradient Boost Tree classification both find the standard deviation of temperature fluctuations to be the most important parameter, which best classify structure loss after 6 hours of body.

Predictive Model

In order to predict structure loss, a binary logistic regression was used for classification of structure loss. Different parameters were used for regression and one parameter provided a high accuracy of classification for ingots that have structure loss after 6 hours of body, namely the standard deviation of the temperature fluctuations around a moving average. This is the parameter that gives best prediction in Sequential Feature Selection. The confusion matrix is shown in Table 2. 99.6% of the ingots in the test set are classified correctly, and 96% of ingots with structure loss are predicted to have structure loss. The binary logistic regression predicts a higher probability of structure loss with reducing standard deviation of the temperature signal. This can be due to absence of manual temperature increases causing less temperature fluctuation and increased frequency of cold melt and structure loss.

TABLE 2. Confusion matrix for classification based on the standard deviation of the temperature signal in the last 85-25 min of ingots with structure loss or for the entire body for ingots without structure loss.

	Actual non-SL	**Actual SL**
Predicted non-SL	2068	8
Predicted SL	2	188

CONCLUSION

After four hours of body (ca. 240 mm), ingots grown with no spikes in heater power due to manual adjustments by furnace operator have a significantly higher chance of structure loss than ingots for which the operator has increased the temperature at least once. This explains why the heater power and temperature fluctuations are lower for ingots with structure loss than ingots without structure loss. Most ingots with structure loss have significantly higher average pull speed than ingots without structure loss, likely due to higher interface deflection and higher thermal stress. A threshold was identified for which there were no ingots with structure loss at lower pull speeds. There is a significant difference in the proportion of ingots with structure loss between two feedstocks. A binary logistic regression was used for classification of ingots with and without structure loss and prediction accuracy on test data was compared for different input parameters. A prediction accuracy of 99.6% was achieved for ingots that have passed six hours of body (ca. 360 mm) by using only the standard deviation of the temperature fluctuations around a moving average in the model.

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