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INNOVATIVE EQUIPMENT FOR THE PREPARATION OF 150MM MULTICRYSTALLINE SILICON

Final Report

CONTRACT NUMBER: FES S/P2/00472/REP

URN NUMBER: 06/906

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INNOVATIVE EQUIPMENT FOR THE PREPARATION OF 150mm MULTICRYSTALLINE SILICON

END REPORT

S/P2/00472/00/REP

Contractor

Crystalox Ltd

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First Published 2006

EXECUTIVE SUMMARY

Objectives

This project had two primary goals; the first to develop the next generation of multicrystalline silicon ingot growth system capable of producing ingots up to 90cm square and weighing up to 500kg. The second goal was to develop equipment which could be used to automate the ingot-to-block processes and to minimise the levels of manual handling required, especially for the heavier blocks of sizes 150mm and above.

Introduction

The photovoltaic (PV) silicon industry has seen a rapid global expansion in recent years with annual growth rates in the order of 30-35%. During this time the majority of the world's solar cells have been made from crystalline silicon either in the form of single crystal or multicrystalline. Crystalox has played a major role in increasing the use of multicrystalline silicon through both the development of equipment for industrial production of multicrystalline silicon and also in the volume manufacture of multicrystalline silicon ingots.

In 2004 the industry standard wafer size was 125mm square, cut from a 66cm square ingot weighing 270kg. However, the drive to reduce cell manufacturing costs has led to the adoption of larger wafer sizes in the range 150 to 156 mm square. The optimum ingot size for the production of twenty five 156mm square blocks requires an 83cm square ingot weighing up to 405kg. The use of this larger ingot increases the weight of usable silicon per run and so offers the potential for reduced production costs. Further R and D is already underway on cell production using even larger wafer sizes - up to 210mm - and in view of this development of a 90cm square ingot size suitable for this block dimension has also been undertaken.

In addition to developing large scale crystallisation equipment, Crystalox has also undertaken the design and manufacture of new downstream ingot processing equipment including systems for block end removal, chamfering, block measurement and final inspection. Previously, procedures to prepare the blocks ready for wafering have largely depended on manual handling to transfer the blocks between process stations and for most operations within each station. The increase in block size from 125mm, through 156mm to 210mm has increased the weight of the block from 9kg to 14kg and 22kg respectively and these larger blocks cannot be manually handled easily or safely. To reduce repetitive handling and the risk of injury and block damage special automated equipment has been developed to replace the current operations.

Work Summary

The overall project has been carried out in two distinct sub-tasks, a) the design and build of the crystal growth system and b) the design and manufacture of block processing equipment.

Task a) Crystal Growth System

A novel crystal growth system was designed to be capable of producing either an 83cm ingot ~400kg weight, optimised for 156mm blocks, or a 90cm ingot, ~470kg, optimised for a 210mm block size. The growth system comprises of four main assemblies: -

- Frame.
- Chamber.
- Furnace.
- Control system.

The frame supports all the major sub-assemblies, and also the majority of the ancillary equipment. The design of the frame enables the chamber to be mounted high up and allows the chamber base to be opened for easy loading and unloading of the ingot. The sealed, water-cooled chamber allows a controllable inert gas atmosphere to be maintained to reduce contamination of the silicon during growth and also prevent the hot graphite furnace components from high temperature oxidation. The furnace components located inside the chamber comprise induction-heated large diameter graphite susceptors which heat the crucibles containing silicon by direct radiation. The outer surfaces of the susceptors are insulated to reduce undesirable heat losses.

The complete crystal growth cycle of melting, crystallisation and ingot cooling are fully automated by means of PC based control software. This control software, which allows fully-programmable automatic control, or manual intervention if required, has been wholly developed by Crystalox.

Task b) Block Processing Equipment

The existing manual ingot-to-block processes were investigated and broken down into separate activities or cells, and each cell was then considered for automation and increased throughput. From this analysis three operations were identified for development under this programme. All equipment was designed to cope with the present 125mm block size through to the potential 210mm size.

- i) Block chamfering.
- ii) Inspection.
- iii) Packing.

i) Block Chamfering

The chamfering process adds a narrow chamfer to the four longitudinal edges of the block to prevent chipping of sharp edges during subsequent handling.

The chamfer machine was designed around a steel hollow section frame which supports the three axes. The action of the chamfering machine has been split into three operations; loading / unloading, block rotation and grinding of the chamfers. A high speed carriage carries the block from the loading position to a position above the diamond grinding wheel. The wheel is mounted on a high precision ball screw which can be moved to give the user a fine control over the size of the chamfer to be ground. After the block has been translated over the grinding wheel and the first chamfer produced the block is lifted from the carriage and rotated to present the next edge for chamfering. This process is repeated three times until all four edges have been chamfered when the block is returned to the unload position. After each chamfer has been ground it is immediately measured and compared to the required specification. If the chamfer is outside specification the operator is alerted.

ii) Inspection

At the end of the block production process every block must be measured and quality checked before it can go on to wafering. There are five individual operations which are necessary: -

- Block length measurement.
- Block width and height measurement.
- Electrical resistivity measurement.
- Labelling to identify the block.
- Visual Inspection for defects.

An integrated inspection machine has been developed which brings four of these five separate steps together. The machine utilizes a through feed belt which transfers a block sequentially from a loading position through three operational stations and on to an unloading position. The three stations carry out in turn, block length and geometry measurement, resistivity measurement and labelling.

A block is first manually loaded at a start position on the feed belt from where it passes to the first measurement station. The first station uses non-contact lasers to carry out both length and geometry measurement. The lasers have been specially selected to work with silicon, as most red lasers used in conventional measurement tools give unreliable readings on silicon which is transparent in the infrared part of the spectrum.

At the second station the electrical resistivity of the block is measured at both ends by means of an inductively-coupled probe. In order to obtain reproducible readings the probe is provided with a touch-sense system which allows the contact force between the probe and silicon to be controlled and precisely repeated.

Labelling is carried out at the third station which prints and barcodes a label with all the required identification and measured data and then attaches the label to one face of the block. The block is then moved to an unload position.

iii) Packing

From the unload position a robot with a specially designed handling tool picks up the silicon block. The robot presents each face of the block to a high resolution camera system which is programmed to recognise and identify cracks. On passing all the required inspection criteria the block is packed by the robot directly into a shipping crate for transfer to the wafering facility. If a block fails it is placed into a reject crate from where it can be retrieved for manual inspection and rework if necessary.

Results

The crystal growth system operated successfully during growth trials and the quality of silicon produced met photovoltaic standard requirements. The testing of the growth system was undertaken through a series of growth trials, each designed to progressively test the system's capabilities. The first trials tested the functionality of the graphite furnace, power supply and coil arrangements; only minor optimisation was required to obtain efficient coupling. Heating trials were then undertaken to prove the operation and robustness of the furnace design. The susceptors and insulation showed no damage or problems at the completion of the tests. Work was undertaken in parallel to assess the effectiveness of a pyrometer for both temperature control and monitoring but the repeatability between runs in the existing set-up was inadequate to allow it be used in production and thermocouples were retained.

The reliability of the large silica crucible was recognised as a potential risk in the development of the new crystal growth system, and the first run with a charged crucible used a relatively low quantity of silicon to minimise potential damage to the furnace components if the crucible failed. No problem was encountered and subsequent trials used a full silicon charge of 470kg in 90cm crucibles and 405kg in

83cm crucibles. Throughout the trials the design and manufacture of the crucibles proved to be robust with no failures or leakage occurring.

The growth programme utilised for the crystallisation runs was based on pre-existing parameters for the Crystalox 66cm growth systems, with a number of changes to suit the larger volume of silicon being processed. The ingots produced displayed both grain size and a crystalline orientation suitable for cell manufacture together with electrical resistivity and minority carrier lifetimes in the ranges required for good cell performance. The initial trials have allowed some preliminary conclusions to be drawn on the yields and throughput of the new system in comparison with existing furnaces. The production cycle time was only marginally longer than the existing 66cm systems and throughput of silicon was greatly improved. Gas and electricity consumption relative to weight of silicon produced were found to be similar and slightly higher, respectively. These initial results are very encouraging and lead Crystalox to believe that further optimisation of the process will provide higher production efficiency.

Operational testing of the block-processing equipment was carried out with two complete ingots cut to produce fifty 125mm x 125mm blocks which were processed under trial production conditions.
The chamfer machine performed extremely well with all chamfers produced to the required size and tolerance. Compared to the existing manual processes the handling required during chamfering was reduced by in excess of 60%, the new machine also having the advantage that all chamfers were identical and individually measured. However, the cycle time required to process an ingot was 25% slower than using manual methods.

The inspection machine and robot packaging cell were tested as a single unit. The through feed system, resistivity measurement and labelling performed well, achieving a cycle time of around 30 seconds per block, within the design specification. However, the accuracy of the laser measurement station did not meet the initial design requirements and the repeatability and accuracy were an order of magnitude worse than expected.

The robot packing cell was a great improvement on the manual packing process and eliminated all manual handling. Although the packaging cell process time was slower than the initial design specification, the cell achieved comparable process time to the existing operations.

The crack detection camera system generally worked well when identifying medium to large cracks, but with small hair line cracks the system did not produce repeatable results.

Conclusion

The project has successfully developed the crystal growth system and block process line as set out in the initial proposal. The use of 3D CAD and finite element analysis (FEA) has been successfully implemented on this project and has greatly reduced the time and costs associated with the development of operational systems by identifying and resolving potential problems before components were put into manufacture.

The new crystal growth system showed that 90cm and 83cm ingots could be grown successfully and within an acceptable cycle time, only slightly longer than the present Crystalox 66cm ingot system. Initial performance of the new system was generally comparable or better than that achieved with the existing systems. The new system needs now to be subjected to a more rigorous test programme within a pseudo production environment to resolve other issues of performance, maintenance and reliability.

The block processing task has produced two operational machines which demonstrate that some of the key parts of the manual process of ingot-to-block production can be successfully automated.

The chamfer machine, inspection machine and automated packing all performed well. In a few areas the processes did not achieve the full performance that was specified at the outset however, with further software and mechanical development the issues of process time and measurement accuracy can be expected to improve.

Experience has been gained in key areas such as the use of robotics, automation and defect detection camera systems and this knowledge will be used to further improve each system with the ultimate aim of constructing a fully-integrated pilot production system.

Further Recommendations

Initial trials on the silicon crystal growth system have been very encouraging but further developmental work under pilot production conditions is desirable to improve the system operation and efficiency including:

- Development of the crystallisation programme to reduce cycle time.
- Development of the pyrometer system to allow furnace temperature control.
- Optimisation of furnace insulation to reduce power consumption.

The block processing machines have been considered successful at this stage but there are key areas that require further work to enhance performance:

- Improved laser measurement accuracy and repeatability.
- Reduction of cycle times for the block chamfering and automated packing systems.
- Development of the defect detection camera system for recognition of finer cracks.

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1) INTRODUCTION

The photovoltaic (PV) silicon industry has seen a rapid global expansion in recent years with annual growth rates in the order of 30-35%. During this time the majority of the world's solar cells have been made from crystalline silicon either in the form of single crystal or multicrystalline. Crystalox has played a major role in increasing the use of multicrystalline silicon through both the development of equipment for industrial production of multicrystalline silicon and also in the manufacture of large scale multicrystalline silicon ingots.

In 2004 the industry standard wafer size was 125mm square, cut from a 66cm square ingot weighing 270kg. However, the drive to reduce cell manufacturing costs has led to the adoption of larger wafer sizes in the range 150 to 156 mm square. The optimum ingot size for the production of twenty five 156mm square blocks requires an 83cm square ingot weighing up to 405kg. The use of this larger ingot increases the weight of usable silicon per run and so offers the potential for reduced production costs. Further R and D is already underway on cell production at even larger wafer sizes up to 210mm and in view of this, development of a 490 kg 90cm square ingot size which matches this block dimensions has also been undertaken.

In addition to developing large scale crystallisation equipment, Crystalox has also undertaken the design and manufacture of new downstream ingot processing equipment including systems for block end removal, chamfering, block measurement and final inspection. Previously, procedures to prepare the blocks ready for wafering have largely depended on manual handling to transfer the blocks between process stations and for most operations within each station. The increase in block size from 125mm, through 156mm to 210mm has increased the weight of the block from 9kg to 14kg and 22kg respectively and these larger blocks cannot be manually handled easily or safely. To reduce repetitive handling and the risk of injury and block damage special automated equipment has been developed to replace the current operations.

2) PROJECT OBJECTIVES

This project had two goals; the first was to develop the next generation of multicrystalline silicon growth system capable of producing ingots up to 90cm square. The second goal was to develop a series of machines to automate the block processing and inspection stages and to reduce manual handling of increasingly large heavy blocks.

2.1) 90cm Multicrystalline Silicon Growth System

This new directional solidification system was specially designed for the production of silicon ingots with dimensions 83cm or 90cm square, the optimum sizes for cutting into industry-standard 156mm and future 210mm square blocks respectively. The project covered all aspects of design and manufacture and addressed the requirements for the enhanced capabilities, including: -

- Furnace hot-zone components.
- Mechanical structural requirements.
- Medium frequency (MF) coil design.
- MF generator power to coil matching.
- Crucible design, manufacture and reliability.
- Crucible handling and preparation.

The use of the latest 3D CAD software in conjunction with finite element analysis (FEA) modelling allowed more accurate predictions and specification of critical components. Enhanced computer modelling also greatly improved the efficiency of undertaking the detailed design and decreased manufacture and assembly times. The new system underwent extensive testing and the design was finally validated by the growth of multicrystalline ingots which were subsequently made into solar cells.

2.2) Process Automation for Larger Blocks

The existing system used by Crystalox requires a large degree of manual handling of the blocks. With a standard ~9kg 125mm square block the handling could be a carried out safely with only manual operations. However the larger sizes of blocks for 156mm wafers (~14kg) and 210mm wafers (~22kg) require a different approach with an increased use of automation to reduce the level of manual handling needed by the operators.

Each individual process in the ingot-to-block production has been assessed for its viability to be automated. As each part of the overall process has its own unique design challenges, the current project is not aimed at automating each step of every process but targets the key stages that would significantly reduce the manual handling of the larger blocks.

Silicon is a fragile, brittle material which can be very easily damaged. The high inertia of 156mm and 210mm blocks calls for specially developed handling and process techniques to be used.

3) 90cm MULTICRYSTALLINE GROWTH SYSTEM

3.1) Specification

The system was designed for the efficient production of suitably sized ingots to allow high yield conversion to both 125 and 156mm blocks as currently required by the PV industry. The implications of a future increase in wafer size to 210mm were also considered.

The design encompassed the following:

- Capability for growth of large ingots with dimensions 83 or 90cm square.
- Multicrystalline silicon produced by the system should comply with the requirements of the PV industry for solar cell manufacture, including:
  - Resistivity within the acceptable range 0.5 to 2.5 cm
  - Average lifetime of blocks >5μs
  - No microcrystallinity, grain size >1mm^2
- Material specifications, design and manufacture must comply with the relevant British Standards.
- The system should be easy to maintain and cost effective in operation.
- Provision to be made for the installation of heavy, delicate furnace components.
- Sealed, water-cooled chamber enabling the use of vacuum (10^{-2}mbar) and controlled atmosphere.
- A fit-for-purpose silica crucible
- Floor level crucible/ingot loading and unloading for simple rapid turn-round.
- Turn-key automatic system control

3.2) Growth System Design

Controlled directional solidification (DS) is necessary for the efficient rejection of impurities, growth of suitably sized grains and for the production of the highest quality silicon. The DS process involves extraction of heat from the base of the crystallising ingot under controlled conditions and this requires careful monitoring and control of a number of critical variables including furnace temperature gradients, crucible translation and the rate of heat extraction from the base of the crucible.

The design called for an elevated stainless-steel chamber to allow loading and unloading to be carried out from floor level utilising a fork-lift truck. The chamber has a double wall design to allow full water-cooling and was dimensioned to accommodate the 90cm square crucible and furnace components.
The existing Crystalox growth system utilises medium frequency (MF) heating and Crystalox has developed this technique extensively for this application.

The overall design comprises the following major sub-assemblies.

3.2.1) Frame

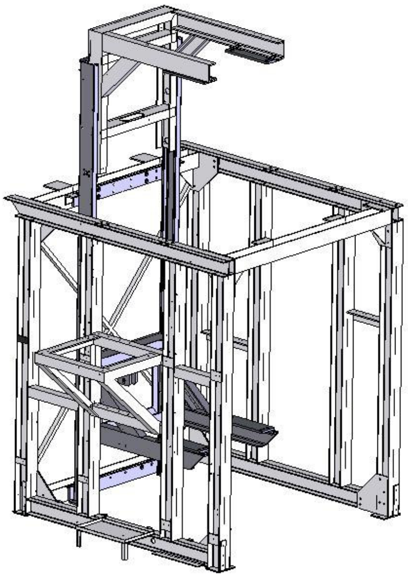

Figure 1 - Frame

3.2.2) Frame Assembly Frame

The frame assembly shown in Figure 1, supports an elevated chamber to allow base opening and facilitate easy crucible loading. The frame is largely constructed using steel universal beams to afford a high strength to weight ratio.

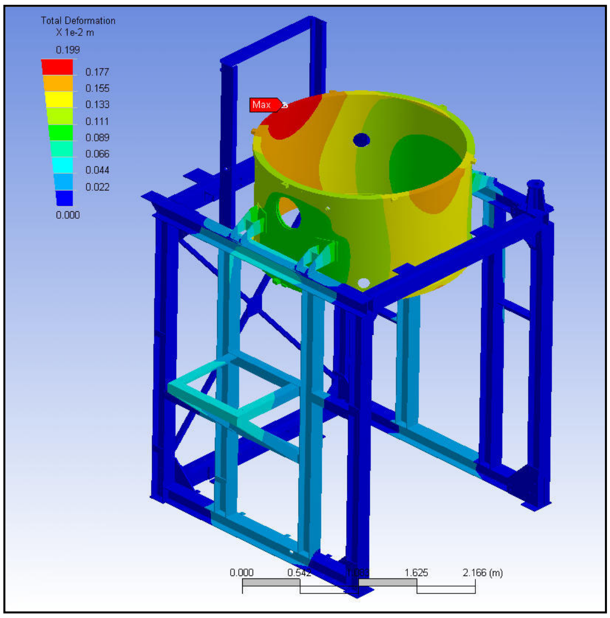

Figure 2 - Frame Stress Analysis with Chamber Body

The frame was analysed using FEA (finite element analysis) Figure 2, to verify the design.

3.2.3) Controlled Atmosphere Chamber

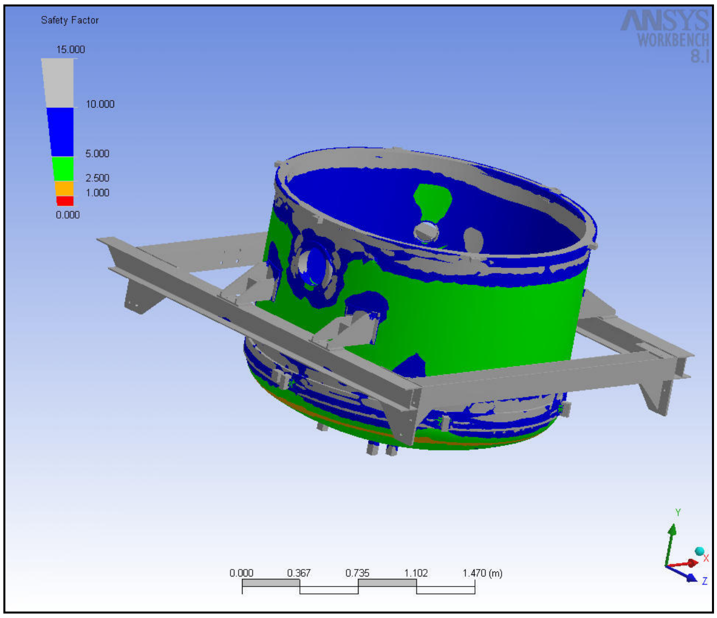

Figure 3 - Controlled Atmosphere Chamber Stress Analysis

The sealed, vacuum compatible chamber was designed to house the furnace and supports. To allow good access for maintenance of internal furnace components and to allow ease of manufacture, the lid and base of the chamber were designed to be removable. The base of the chamber provided access for loading and unloading of the crucible and ingot. The stainless steel chamber was double skinned to allow efficient water cooling and this in conjunction with the pressure rating has necessitated the design to comply with international standards relating to pressure vessels. FEA stress analysis, Figure 3 was used to enable potential stress centres to be eliminated prior to manufacture.

3.2.4) Furnace Design

The furnace assembly is constructed using commercial monolithic high-purity graphite together with carbon bonded carbon fibre insulation components. The graphite susceptors are induction heated which in turn heat the silicon principally by radiation at the high temperatures required.

The high purity graphite susceptors used in the furnace were much larger than used previously. Special handling techniques were developed to allow for transporting the components to our facility and their safe installation.

3.2.5) MF Coil Design

The design of the high conductivity water-cooled copper coil was critical to the successful and economic operation of the furnace. In order to achieve a high coupling efficiency between the MF generator and susceptors Crystalox has worked closely with the MF generator supplier to understand the critical parameters set within the generator which has allowed the effective matching of the coil and generator to give the most efficient power usage.

3.2.6) Temperature Measurement

A pyrometer was specified to control the furnace temperature to avoid problems associated with thermocouple drift experienced at these operating temperatures when using Platinum/Rhodium thermocouples. The additional capital cost of a pyrometer was felt to be justified by the anticipated improved stability and reduced maintenance requirements. A pyrometer can be used at some distance from the target and this enables its location to be outside the chamber, simplifying the design when compared to the use of thermocouples.

3.2.7) Crucible Design

The crucible is used to contain the silicon feedstock and defines the geometry of the finished ingot. The new crucibles, manufactured from slip cast fused silica to withstand the high operating temperatures and to be compatible with molten silicon in terms of purity and retention of mechanical stability, have been developed in conjunction with commercial suppliers. The design of the crucible, especially in conjunction with the crucible support graphite inside the furnace, is critical to obtaining reliable performance.

3.2.8) Machine Control Software

The new control software is based on that employed by the present Crystalox 66cm growth system, which has been modified specifically for the new system. The software and control system allows ingot production to be carried out totally automatically, but also allows full operator access during a run a feature that is critical during the early development phase of the project. The software automatically controls the process of melting, growth and cooling. A number of significant changes in the software have been implemented to improve the control of the crystal growth process and allow easy set up for changes in the growth parameters required for the different ingot sizes.

3.2.9) Loading /Unloading Operations

A specially modified loading truck, based on a commercially available product is used for loading and unloading the crucible into/from the furnace. The increased weight, inertia and momentum of the loaded crucible has necessitated the use of dampening buffers to prevent shock damage to both growth system and crucible during the loading process.

3.3) Growth System Build

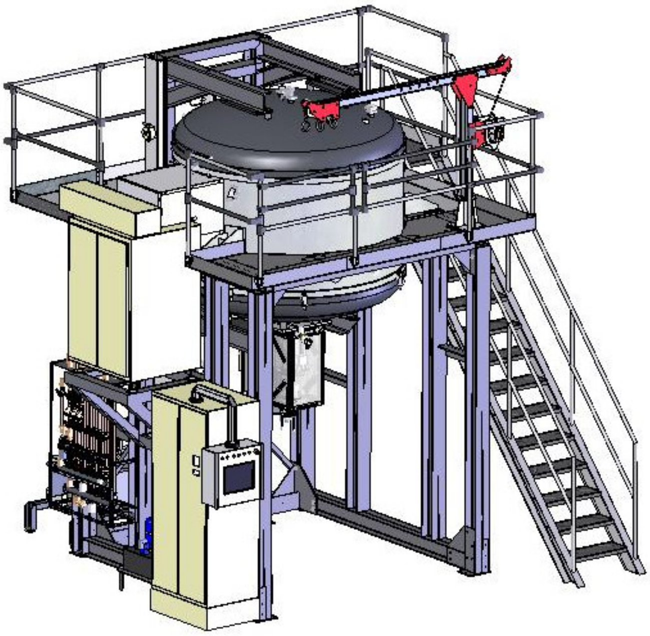

Figure 4 - Multicrystalline Growth System

The manufacture of many of the mechanical components and sub-assemblies was undertaken by a variety of sub-contractors, with all parts and sub-assemblies fully tested at Crystalox before final assembly. All electrical and software development was carried out in-house.

3.4) Growth System Testing

3.4.1) Test Programme

The testing of the 90cm ingot production system, Figure 4 included checking and optimisation of all the operational systems and sub-systems. The test programme addressed the following:

- Chamber lid operation - used for furnace installation and maintenance.
- Chamber base operation - used for crucible loading.

- Commissioning of the water cooling system including flow rates, alarm levels and interlocks.
- Vacuum integrity of the chamber and vacuum pumping system.
- Operation and calibration of the inert gas flow system.
- Operation of the crucible support translation/rotation system.
- Set up and calibration of the temperature control and monitoring systems.
- Set up and testing of all safety and control interlocks.
- Operation of the MF generator and control systems.
- Set up and operation of the supervisory control and data acquisition (SCADA) system.

Following full testing of individual sub-assemblies, the performance of the complete system was verified during controlled heating and cooling and in silicon growth trials.

A test sequence was established to confirm the operation and reliability of each system component. For example

- The system was heated to test for effective MF power coupling.
- High temperature capability.
- Stability of closed loop temperature control.
- Pyrometer and thermocouple operation.
- Controlled cooling of the system.

Silicon growth runs were then carried out, using low-grade silicon feedstock and low charge weight as a means of checking the reliability and high temperature strength of the larger crucible design. Crucible failure and leakage of molten silicon was a concern and systems were put into place to limit furnace damage in such an event and although the growth cycle was fully automatic all initial runs were carried out under manned supervision. Further runs were carried out using PV-quality silicon feedstock for characterisation. Ingots were sawn into blocks to check for grain structure, inclusion levels and to allow resistivity measurement.

3.4.2) Assessment of Silicon Quality

The assessment of silicon quality included a study of both mechanical and electrical properties.

a) Mechanical properties.

The shape of the solid liquid interface is important in the segregation of impurities by the DS technique and impurities rejected to the top of the ingot may be removed by sawing with no significant loss of yield. A visual inspection of blocks cut from an ingot allows assessment of crystal structure including the solid/liquid interface shape, growth direction, grain size, grain boundary orientation and undesirable inclusion levels. Each of these parameters can affect cell performance. Stress-free silicon is required, achieved through correct annealing of the ingot to avoid stress-cracking and loss of yield during the blocking process. High levels of hard inclusions can also cause difficulties when cutting the ingots into blocks or wire breakage during wafering, leading to lower wafer yields.

b) Electrical Properties

Minority carrier lifetime and resistivity are industry-standard checks for photovoltaic grade silicon. Facilities are available for both these measurements. These parameters can be affected by a number of variables including feedstock quality, metals content and other impurities diffusing into the ingot from the furnace materials, crucible / coating and/or the growth atmosphere.

3.5) 90cm Growth System Results

3.5.1) Crystal Growth

All the test runs described above operated were completed successfully, with only moderate adjustments being required to achieve the desired results. The larger crucibles proved reliable throughout the trials and assessment and measurements taken on blocks cut from ingots produced using PV-grade feedstock showed the required crystal structure and electrical properties necessary for manufacture of solar cells. However, some problems with operation of the pyrometer were encountered, as described in Section 3.5.3 below. Early ingots also had surface deposits and possible causes were identified as process conditions, the quality of feedstock used or contamination of the furnace. Gas flow rates were adjusted in later runs producing ingots with cleaner surfaces.

3.5.2) Material Properties

After each run using PV-grade feedstock the ingot was cut to allow measurement of minority carrier lifetime and resistivity and assessment of grain size. Low lifetime can result from impurities in the silicon feedstock or contamination of the melt from the furnace. Minimum average block lifetime was specified as >5µs and two blocks from each ingot were tested.

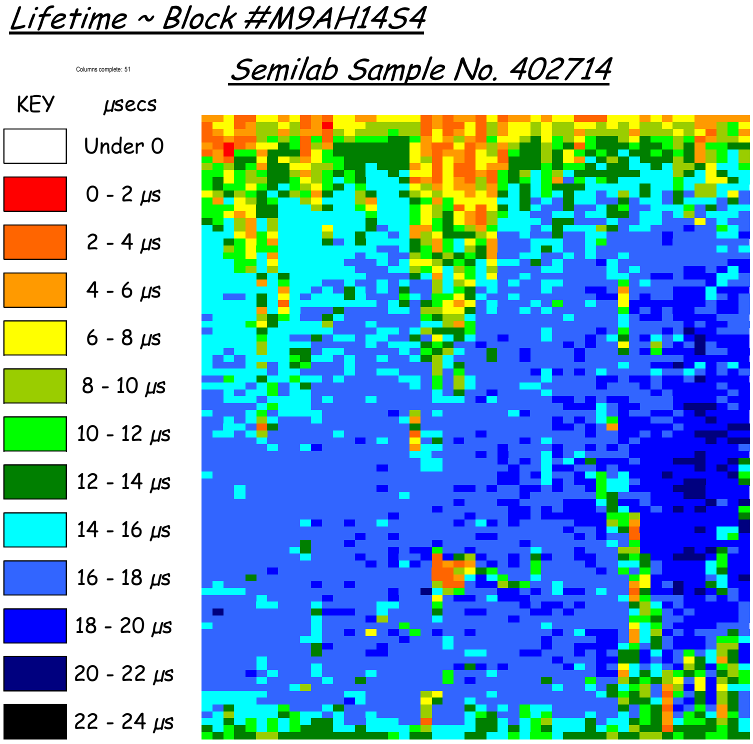

Figure 5 - Lifetime map of a Ingot Block

Figure 5 shows a block lifetime map from a 90cm ingot produced by the system. All blocks tested showed lifetimes exceeding the specification, having average lifetime >10µs, and were similar to typical results obtained from standard production.

The resistivity of blocks was also measured at three points, top, middle and bottom. Control of resistivity is achieved through calculated addition of boron dopant, taking into account impurity levels and the resistivity of feedstocks used. Blocks had resistivity within the acceptable range 0.5 - 2.5 cm, measuring 1 - 1.3 cm.

Figure 6 – Seventh Run; Ingot Grain Structure

Visual inspection and assessment of crystal structure showed the desired highly-oriented, vertical growth with grain cross-sections up to 3 – 4 cm^2. No areas of microcrystallinity (small grain, <1mm^2), which can cause low cell efficiency, were evident, Figure 6.

3.5.3) Pyrometric Temperature Control and Monitoring

The development of pyrometers to replace thermocouples in the operation of this system met with mixed success during the growth trials. Problems with obscuration of the thermowells were encountered and caused unpredictable drift in the measured temperature. The situation was improved using sleeves and transparent rods but results were still not sufficiently stable to offer reliable pyrometric control. As a result the use of thermocouples has been retained for temperature control and monitoring.

3.5.4) Yields

At present the standard Crystalox 66cm ingot production system produces ingots for cutting into industry-standard block sizes of 125mm, 156mm and the future 210mm square wafers though ingot-to-block yields vary depending on the block size.

Development of the 90cm ingot growth system was driven by the requirement to produce the larger block sizes more efficiently and cost effectively. The success of the new larger ingot production equipment will be judged on a number of results, key factors being geometric block yield and silicon quality. Other parameters, including capital cost, space requirement, reliability, spares and utility consumption must also be taken into consideration.

This development programme has shown that this novel 90cm system has the capability for successful and consistent ingot production. Some comparisons between the 90cm ingot growth system and the current 66cm growth system are made below.

The following tables compare the 90cm, 83cm and 66cm size ingots for key production factors.

- Number of blocks produced / ingot.
- Production rate (blocks / hour).
- Power consumption (kWh / block).
- Inert gas consumption (litres / block).

1) Blocks produced per size of ingot

	Number of blocks produced / ingot		
Block size	**66 ingot**	**83 ingot**	**90 ingot**
125mm	25	36	(36)
156mm	16	25	(25)
210mm	9	9	16

Figure 7 – Number of blocks / ingot size

Figure 7 shows the size and number of blocks produced per ingot. As can be seen, the 90cm ingot size is required for cost-effective production of the largest 210mm block.

2) Production Rate blocks per hour, (Cycle time defined as 'run time from start to unloading at 400°C')

	90cm Growth System	
Block size	**83 ingot**	**90 ingot**
125mm	+32%	
156mm	+43%	
210mm		+51%

Figure 8 – Production rate (blocks per hour) compared to standard 66cm production

As shown in Figure 8 significant block production rate increases were realised.

3) Power consumption

	90cm Growth System	
Block size	**83 ingot**	**90 ingot**
125mm	-37%	
156mm	-26%	
210mm		-17%

Figure 9 - Efficiency kWh per block compared to 66cm system

Power requirements / block are greater than the current 66cm growth system, Figure 9.

4) Inert gas requirement

	90cm Growth System	
Block size	**83 ingot**	**90 ingot**
125mm	-9%	
156mm	-1%	
210mm		+6%

Figure 10 - Efficiency inert gas (litres) used per block compared to 66cm system

The efficiency of inert gas use is lower for the 125 and 156, but higher for the 210mm block size, Figure 10.

These early results from initial production trials on the large scale 90cm growth system provide some indication of the advantages of producing larger ingot sizes. Silicon production rate is greatly improved although electricity consumption is somewhat worse and gas consumption broadly comparable with the standard Crystalox 66cm growth system, based on number of blocks produced. The results are encouraging and further process developments can be expected, resulting in ever more efficient production of multicrystalline silicon.

3.6) Conclusion

The project has demonstrated the successful development of an ingot production system capable of growing larger size up to 90cm square silicon ingots suitable for cutting into new larger sized blocks. A number of new areas have been developed and tested including:

- The design and use of larger sizes of high purity graphite furnace components.
- The development of larger crucibles capable of withstanding the increased stresses associated with larger dimensions and increased silicon charge weight.
- Efficient coupling of a new MF power system with larger diameter coils and furnace design.
- Development and optimisation of new control systems.
- New advanced designs to ensure high reliability and reduce manufacture and operational costs.

The ingot production growth system that has been designed and built under this project proved to be at the stage from which it can be further developed during operation. Following process optimisation and proving under production conditions it will be possible to assess the performance of this experimental system with the expectation of having a fully tested and proven design available to meet the future needs of the global PV market.

3.7) Recommendations for Further Research and Development

Initial trials on this larger-scale, 90cm-ingot growth system have been considered successful, with the system proving capable of producing high quality silicon ingots, suitable for use by the PV industry. However further work is necessary before the system can be used for pilot industrial scale production, including:

- Optimisation of the production process to improve the quality of the silicon. Increases in minority carrier lifetime and useable product can be expected, resulting in higher block and wafer yields.
- Development of the hot zone furnace components used, with the aim of increasing efficiency in a number of areas. These include shortening of cycle times to increase productivity, reducing process gas usage and minimising electrical power consumption.
- Further research into the use of pyrometric temperature control and monitoring, to overcome the problems encountered during the trials undertaken to date. Pyrometry remains an attractive alternative to thermocouples offering the potential for higher-reliability and improved long-term stability of temperature measurements.

4) BLOCK PROCESS LINE EQUIPMENT

4.1) Block Processing – Areas for improvement

The process of converting an ingot into blocks ready for wafering can be broken down into several steps which can be considered for automation. The scope of this project was to identify key areas for improvement and to develop these block manufacturing processes.

Three key areas were identified as having the greatest potential for reducing manual handling of blocks and increasing throughput, as follows:

- Edge Chamfering.
- Block Inspection.
- Packaging Cell.

4.2) Edge Chamfering

To protect the edges of silicon blocks, a chamfer is ground along the four longitudinal edges. The chamfers reduce the risk of damage to the block throughout subsequent processing. Chamfering is carried out on a full length block prior to having the top and bottom removed.

Design Specification:

- Chamfer angle of $45^{\circ} \pm 5^{\circ}$.
- Chamfer dimensions 1.0 to 2.0 mm ±0.5mm.
- Throughput of one block / minute.
- To accommodate block sizes from 125mm square to 210mm square.
- Block length up to 385mm.
- To accommodate block weights up to 33kg.
- The machine to be designed ready for automated load / unload.
- The process to use water as the cutting coolant.

4.3) Block Inspection System Specification

The following operations are carried out during the inspection of blocks prior to packing:

- Measurement of block length.
- Geometry checks.
- Resistivity measurement.
- Block Labelling.
- Visual Inspection for defects.

An automatic inspection machine has been designed and manufactured for the first four stages shown above.

4.3.1) Block Length and Geometry Measurement

The block length determines the number of wafers that can be cut from a block and the geometry check ensures that the size is within tolerance and the block is square. Block length is dependent on a number a variables including ingot height, the length of low quality silicon removed from the top and bottom of the block and occasional re-ending necessary to remove any damaged material unsuitable for wafering.

Design Specifications:

- Accommodate block sizes in the range 125mm to 210mm square up to 300mm long.
- The maximum block weight of 25kg.
- Throughput of one block / minute.
- Measurement accuracy: ±0.02mm.
- Measurement repeatability: 0.005mm.
- A minimum of three linear sets of measurements spaced along the length of the block.
- Operating temperature range 5°C – 30°C.
- System to allow simple daily re-calibration with a reference block.
- Data output to a central computer.

4.3.2) Resistivity Measurement

Both ends of each block must be checked to ensure that the resistivity is within the required specification.

Design Specification:

- Accommodate block sizes in the range will be 125mm to 210mm square up to 300mm long.
- The maximum block weight up to 25kg.
- Throughput of one block / minute.
- Data output to a central computer.
- Resistivity measurement range 0.5 cm – 2.5 cm.
- Measurements to be made on both ends of the block.

4.3.3) Block Labelling

All blocks are labelled with a bar code/serial number for identification and traceability. Labels are attached to the top faces of blocks so that visual checking of the four side faces, which are more likely to show signs of defects or damage, can be carried out. A system that could automatically attach a printed label to the top of each block with a throughput of one block/minute was required.

4.4) Visual Inspection/Packing Cell Specification

The current inspection techniques mean that the blocks are manually handled for much of the time, with operators visually checking all faces and edges for cracks or defects. However this project has enabled development of camera image based inspection. To accommodate the camera system effectively it has been integrated into the packing cell rather than the inspection cell. The use of a robot in the packing cell has allowed greater flexibility in the way the camera system has been designed and used.

Once the blocks have passed inspection they are loaded into a crate prior to transfer to a wafering facility. The packing cell was designed to enable:

- Packing of a complete set of blocks from an ingot per crate.
- The capability of ordered packing of blocks from several ingots into multiple crates.
- Facility for rejected blocks.

4.5) Chamfer Machine Design

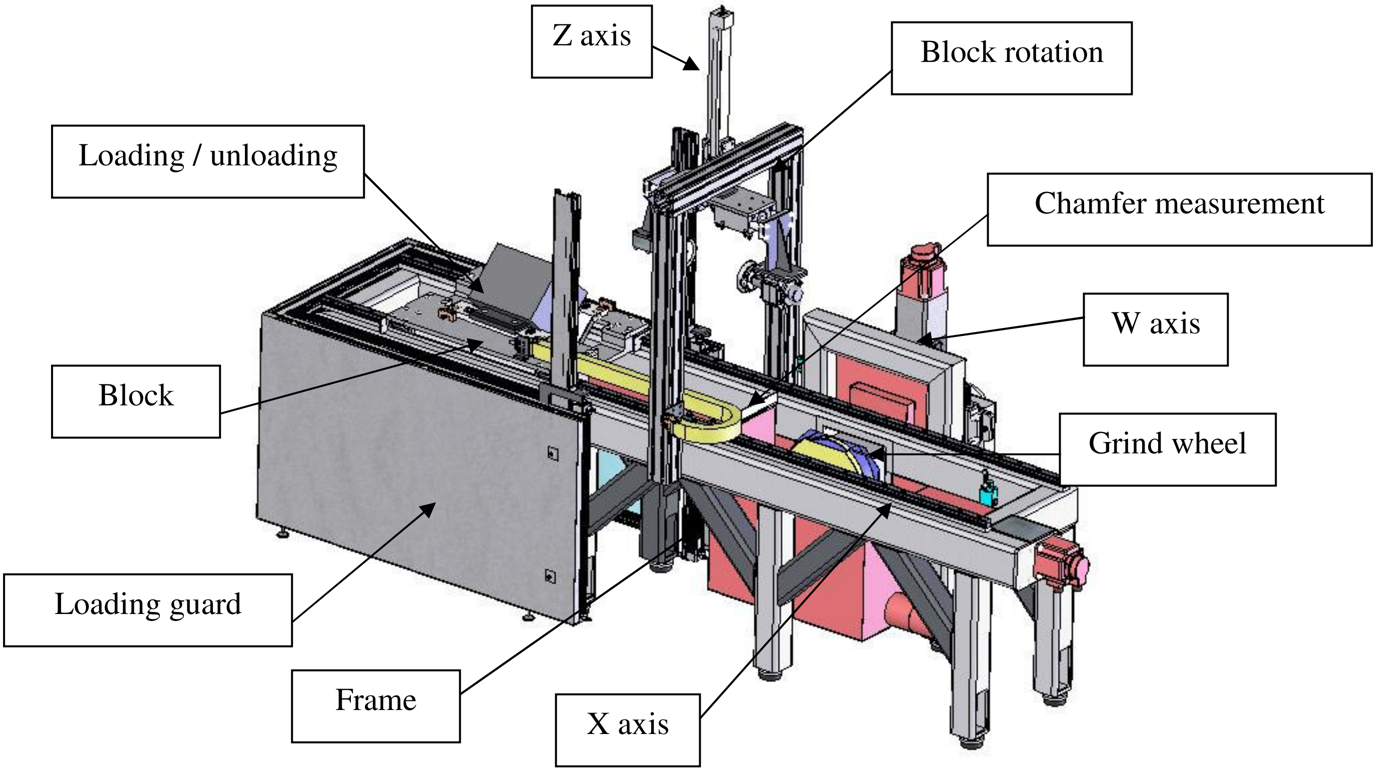

Figure 11 - Chamfer Machine Layout (rear guarding removed)

4.5.1) Chamfering Operations

The chamfer machine is an automated unit capable of grinding a 45 degree chamfer onto each of the long edges of a silicon block and is shown diagrammatically in Figure 11.

The operating sequence for the chamfer machine is as follows:

- A block is loaded at the start position.

- The block travels along the X axis to the grinding wheel, where the chamfer is produced.
- The block travels back to the rotation unit.
- The chamfer is dimensionally checked.
- The block is rotated through 90° ready for the next chamfer.
- The process is repeated to produce each chamfer in turn.
- After all four edges have been chamfered the block is returned to the loading/unloading area.
- If any of the chamfers are outside specified tolerance the operator is alerted.

4.5.2) Frame

The frame was constructed from hollow section steel to provide a high rigidity, stable platform suitable for mounting both the grinding wheel axis (W) and block carrying axis (X).

4.5.3) Grinding Wheel

The hardness of silicon meant that a diamond grinding wheel was required for the chamfering process. The selection of grit size and choice of peripheral speed of the grinding wheel were found critical in achieving satisfactory results.

4.5.4) Cutting Fluid

Clean water is used as a coolant during the grinding process, also reducing the level of airborne dust produced. The water and ground silicon fines are drained and collected in a catchment tank mounted inside the framework.

4.5.5) Grinding Wheel (W) Axis

The chamfer size is programmable and is determined by appropriate positioning of the W axis. Preloaded linear rails were used to support the axis and translation unit

This was driven by a very low backlash ball screw and motor drive system and the speed controlled by a high precision servo drive.

4.5.6) X Axis and Block Support Carriage

A ball screw and servo system was used to move a carriage supporting the block from the loading area to the grinding/measurement/rotation areas along the X axis linear guide rails. The carriage was constructed to support the block so that the faces were at a 45° angle to the horizontal grinding wheel and comprised two separate inner and outer sections. The outer section of the carriage provided strength, whilst the parts which were in contact with the block were made from a compliant material, to minimise damage to the block during loading / unloading. The inner section of the carriage was angled to the drive axis to give maximum coverage of the width of the grinding wheel and thus prevent uneven wear.

Vacuum cups were positioned at each end of the carriage and were used to clamp the block to the inner section during the grinding process.

4.5.7) Block Rotation

The block was required rotate after each chamfer was completed to present the next edge. The block was clamped in position using compliant cups and then raised by a linear drive unit (Z axis) and rotated before being lowered into the carriage to present each edge.

4.5.8) Chamfer Measurement

Measurement was carried out by an array of displacement sensors which were placed onto the block at the completion of each chamfer. If a chamfer was found to be out of specification an alarm sounded so that an operator could assess the problem and make any necessary adjustments.

4.6) Inspection Machine Design

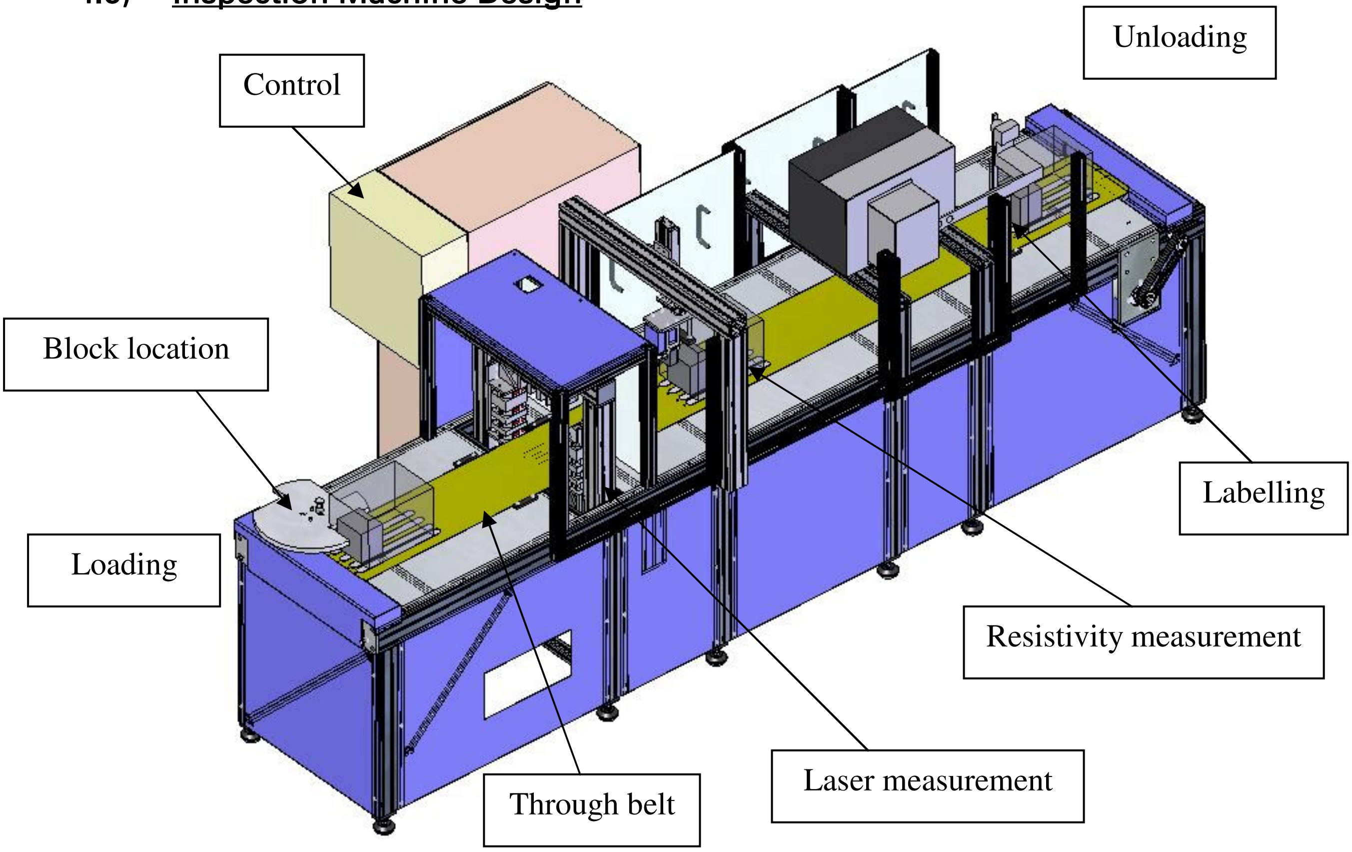

Figure 12 - Inspection Machine

4.6.1) The Inspection Process

The inspection machine, a schematic of which is shown in Figure 12, was designed to automate the process of silicon block inspection and measurement. The system operated in a series of start-stop steps as follows:

- The block was loaded onto a transfer belt at the loading station.
- The block was transferred though the laser measurement station, where the cross section (size and squareness) and length of the block was determined during the transit.
- The block transferred to the resistivity measuring station, where top and bottom faces of the block were measured.
- Meanwhile the next block was loaded at the loading station.

- Following resistivity measurement, the block was transferred to the labelling station where a label was printed and attached.
- The block was transferred to an unloading position where it could be removed either manually or automatically using a robot.

4.6.2) Framework

The framework was constructed from aluminium extrusion. This material enabled a fast and cost effective build with minimal machining required. The frame was designed to allow additional modules to be incorporated should further automation stages be required in the future.

4.6.3) Dimensional Measurement using Lasers

Block size, squareness and length measurements are required for quality control purposes and both contact and non-contact methods were considered. The decision for a non-contact system was based on the inherent advantages including reduced risk of damage to the block and the ability to measure different block sizes without adjustment or recalibration.

Conventional laser measurement systems commonly use red light sources for detection; however, these can lead to unreliable readings on multicrystalline silicon which is transparent in the near infrared part of the spectrum. After extensive collaboration with a laser supplier, a new system was successfully developed for use with silicon providing greatly improved reliability of results. The lasers were mounted in groups of four on each side of the block allowing a calculation to be made of the dimensions and squareness. The block length could also be calculated from the transit time of a block with detection of the leading and final edges of the block.

4.6.4) Resistivity Measurement

The resistivity measurement was made using an inductively-coupled probe. The probe tip was placed in contact with and perpendicular to the silicon surface to ensure an accurate and repeatable reading. The block was clamped beneath the probe head and then the probe lowered from the parked position above the block to the measurement position at the front end. The touch sensitive measuring unit then moved forward until it contacted the surface of the block. The floating head allowed perpendicular self alignment of the probe tip with respect to the block surface.
On completion of the measurement the probe returned to the park position and the block was moved forward to allow access to the back face. The probe was rotated through 180 degrees and the measurement process repeated with both measurements logged and checked against the specification.

4.6.5) Labelling

In order to identify the block it was necessary to provide it with a secure wash and etch-resistant label applied immediately prior to packing. A single label shows details of block length, serial number, date and operator. The label was also used to identify any reject block and the cause of rejection.

4.6.6) Control

The system was controlled by a PC-based human machine interface (HMI). Initial information such as ingot and block number was keyed in as each block was loaded. The measurements were stored for each block to enable generation of packing lists and statistical information.

4.7) Packing Cell

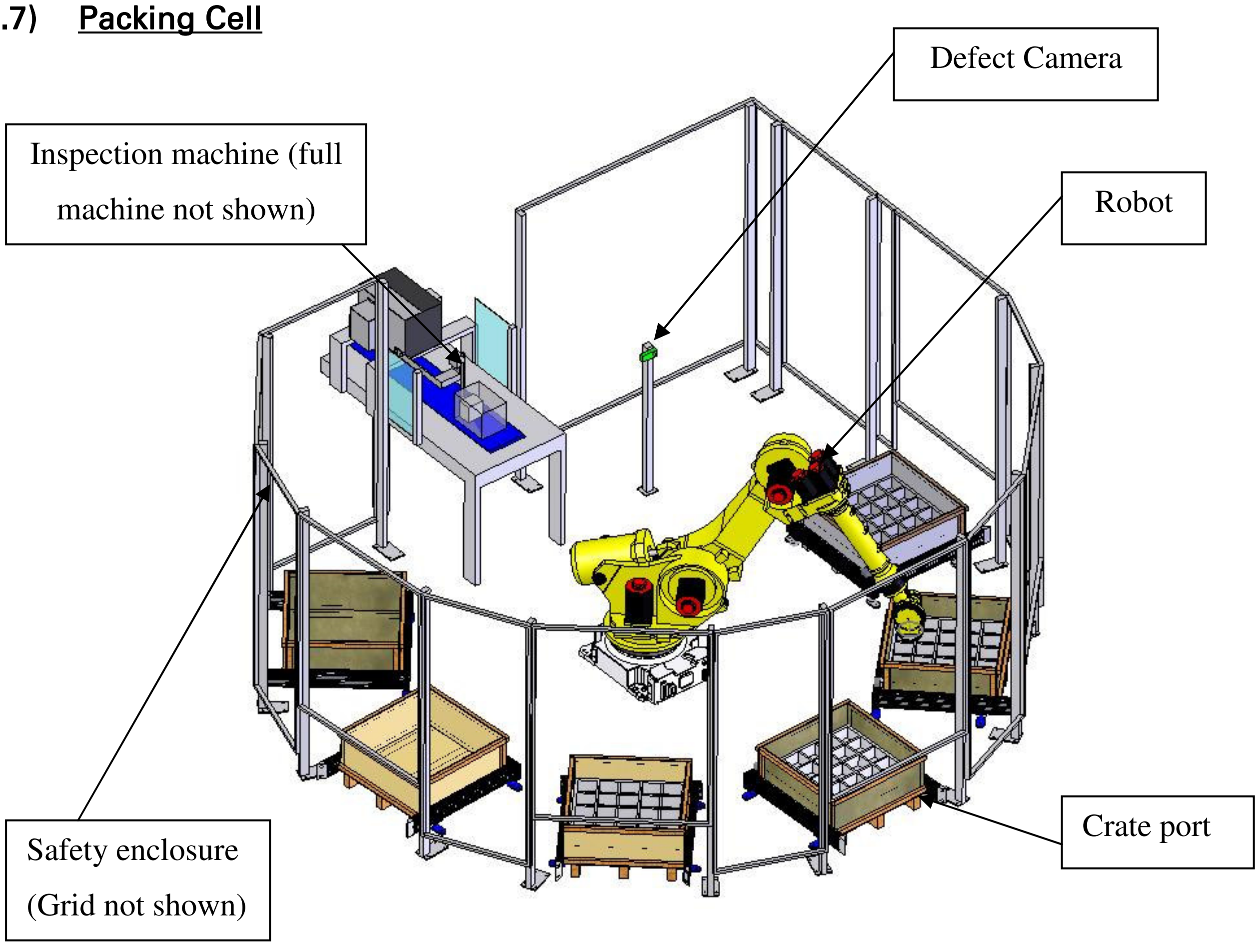

Figure 13 - Robot Packing Cell

4.7.1) Block Packing

The automatic packaging cell, a schematic of which is shown in Figure 13, has been designed to improve efficiency and reduce manual handling of blocks. This packing cell was also fully controlled by the HMI software used by the inspection machine, and thus allowed full integration of both systems.

The robot was used as follows:

- Turn the block through 90° and then pick up from one end face.
- Present the block to the defect-detection camera, one longitudinal face at a time.
- Place the block into the appropriate crate.

The crates are removed and replaced manually from outside the cell.

4.7.2) Robot

A fully articulated six-axis robot was used for this application. The purpose developed vacuum-operated end effector allowed full manipulation of the block from the unloading position to the crate and was integrated into the control system of the inspection machine.

4.7.3) Defect Detection Camera

A final inspection of the block for any damage caused during handling or inspection is desirable prior to packing. To carry this out, a camera-based defect detection system was developed. The robot was programmed to present each face in turn to the camera and high intensity light source. Software was developed to identify cracks or chips but ignore characteristic grain boundaries.

4.7.4) Crate Port

The robot is surrounded by crates (in the prototype cell six crates were used). During testing, it was desirable to pack each block in the same position and orientation with respect to its original position within the ingot. Of the six crate positions available in the cell, four were used for packing of the finished blocks and two were assigned to hold any rejected blocks for manual inspection and re-work. The position and identity of each crate in the cell was automatically read by a sensor.

4.8) Results of Equipment Trials

4.8.1) Chamfer Machine

After initial testing of the chamfer machine a full test was carried out by passing fifty 125 mm blocks (from 2 ingots) through the machine in a trial ‘production’ run. Chamfers were successfully produced and measured and the system operated without any block handling difficulties. Manual block manipulations and the risk of handling damage have been significantly reduced, meeting the project requirements. However, at present the process time has been increased by approximately 25%.

4.8.2) Inspection Machine & Packing Cell

The inspection machine and packing cell were tested as a single unit. To test both systems, fifty 125mm square blocks were processed. In order to test the defect camera two blocks with cracks were processed.

The inspection machine performed well with a process time for each block being approximately 30 seconds, including operator time required for entering block information, so fulfilling the initial design criteria.

The accuracy of dimensional measurements achieved using the advanced lasers and software utilised was +/-0.2mm with a repeatability of 0.08mm, comparable with current manual methods. However, the initial specification required the measurement to be an order of magnitude lower and further work is required to improve the accuracy and repeatability.

The resistivity system measured each block accurately and with a high degree of repeatability. Similarly the labelling system worked reliably.

The defect detection camera generally worked well, successfully identifying larger cracks. However, some of the smaller hair line cracks were more difficult to distinguish. Overall the system operation was considered to be a success, and met the majority of specification targets.

The packing cell was an improvement on the manual packing process and eliminated the requirement for manual handling. The process time, however, did not fully meet the performance targets. The desired specification for the whole process of pick-up, inspection and packing was one block / minute. In operation, the time taken was between 72 and 82 seconds depending on the distance to the packing crate. Although the cycle time has not met the project goal it was still at least comparable to the present manual process.

4.9) Conclusion

The project has produced two operational development machines and both machines demonstrated that key manual processes could be successfully automated. The machines have been thoroughly tested and showed robustness of design and manufacture and demonstrated effective operation, the only criticism concerning speed of operation in some areas. Overall, the systems were found to perform well, with only minor modifications required to meet the majority of specification targets.

These new machines will now be subjected to more rigorous testing to further identify issues such as operating yield, repeatability, quality and reliability before the next stage design improvements can be considered. It is anticipated that once these systems have been thoroughly tested and developed, the interlinking of these and other modules would allow the assembly of one continuous automatic production line. It is this ultimate goal of the company to have such a production line developed to allow future increases in production capacity to be undertaken in a more cost effective manner with the aim of significantly reducing production costs.

4.10) Recommendations for Further Research

The development of these process machines has been considered a success, but at this stage there are areas which could be improved by further work in terms of reducing cycle time and improving measurement accuracy. The following areas have been identified as those requiring further development:

- The geometrical measurement (cross section) lasers require further software development to improve their accuracy and repeatability.
- The defect detection system only recognises larger sized cracks at present; with further development of hardware and software the detection of fine cracks is anticipated.

- Development of a more stable and secure robot end effector which supports the silicon block during acceleration and deceleration would allow the robot to operate at higher speeds so reducing cycle time.
- The chamfer machine cycle time needs to be reduced to meet the desired throughput levels.

ACKNOWLEDGEMENTS

The author acknowledges the help and support of all Crystalox Staff leading to the successful conclusion of this project and to the DTI for financial assistance.