

CASTING AND COOLING/CRUSHING OF SILICON METAL AND SILICON ALLOY AT BÉCANCOUR SILICON INC.

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ABSTRACT

Casting, cooling and crushing/sieving of ferroalloys has followed a well-established pattern in the whole ferroalloys industry for many years. Usually liquid metal or alloys are cast into flat open moulds of various shapes and forms, and after solidification the product is removed from such mould to further cool down to a temperature where subsequent handling steps are feasible. Common to all mechanical product fractioning is the generation of dust and fine size material with lower product value.

For almost 10 years, Bécancour Silicon has investigated alternative roads to improve technology and/or reduce or solve problems related to those disadvantages. Since 1995 Bécancour Silicon operates a special silicon granulation plant targeted to product for the chemical treatment/conversion of silicon for silicones production.

Prior to the development of the granulation process, various systems had been evaluated, among others the vibrating casting bed, now in operation elsewhere but judged inappropriate because of the tapping volumes to be handled at Bécancour Silicon and other disadvantages.

After evaluation of all aspects of heat transfer, radiation, and conductivity in relation to transfer media and within the product, a novel system was designed and built.

1. INTRODUCTION

The traditional method of casting silicon metal consists of emptying the liquid silicon holding ladle into shallow open cast-iron moulds or cast beds, usually lined with a layer of fine silicon metal to firmly insulate and contain the liquid silicon in shape until it is frozen. The layer of fine sized silicon further facilitates stripping of the solidified cake. For other than the lowest quality commodity grades, fines are preferably of the same chemical composition as the liquid silicon in the ladle to provide for a consistent chemical analysis and prevent contamination from the fines layer that is remolten and forms part of the new cast. In cast iron mould application, one usually removes the solidified cake from the mould as soon as possible to avoid mould heating and get the mould ready for the next cast. Before the solidified cake can be further processed through crushing and sizing devices, it has to be cooled down to almost ambient temperature to allow safe handling and transporting on conveyor belts or other temperature-sensitive equipment.

Depending on the customer's further use of the silicon, various size specifications in place complement the chemical specifications, resulting in a product matrix for the producer. The aluminum industry, as one major consumer of silicon metal, typically dissolves solid silicon lumps in or by liquid aluminum, thereby slowly dissolving the specifically lighter silicon in the specifically heavier aluminum. The important criteria are silicon recovery and dissolution time. To avoid that silicon floats on the surface of liquid aluminum thereby getting trapped in the surface slag, the aluminum industry usually sets a limitation to the quantity of smaller size particles. But since any mechanical fractioning of silicon – due to product-inherent friability – generates fine sizes and dust, these inferior sizes downgrade a product that is chemically 100% suitable. Each silicon producing plant has its own recipes to minimize this economic impact.

2. OBJECTIVES OF OUR DEVELOPMENT WORK

Since the early '90s, a lot of systematic work has been done at *Bécancour Silicon (Silbec)* to understand the principles of heat exchange between liquid silicon and a surrounding medium, between solidifying and solidified silicon, and finally of solidified Si, at temperatures from 1400°C down to 300°C, as well as the heat transfer limitations from silicon to the medium, and the heat transportation within the silicon. This work enabled us to design and build a water granulation plant converting liquid silicon into a structure optimized small granule for its application in the chemical industry, in particular for the conversion into methylchlorosilanes[1]. However, this product cannot satisfy the aluminum industry, even if it would be well qualified as regards homogeneity. In order to cope with the increasing demands to find more efficient and thereby less costly ways to convert liquid silicon into a product physically suitable at the maximum percentage for – or even desirous to – the aluminium industry, our work and that of others published in the early '90s, including the evaluation of the vibrating copper plate[2] as a rapid cooling device, was re-evaluated[3].

Therefrom the following requirements were postulated:

- Casting straight from the ladle into the machine without using runner or intermediate guides.
- Maximum casting time of 20 minutes for the highest volume of silicon metal that is received from the biggest of *Silbec's* three furnaces.
- Re-availability of the machine for the next cast within maximum 40 minutes after the previous one.
- Appropriate cast thickness so that no pre-crushing and a minimum of jaw crushing, if at all, is required to achieve standard aluminum sizes.
- No labour-intensive cleaning or preparation of the casting machine between casts.
- Potential for sequence casting using metal from more than one furnace without overloading the machine, neither thermally nor volume-wise.
- Clear separation and distinction of different metal grades when handling casting from more than one furnace.
- Minimization or avoidance of any wear and tear to the heat exposed surfaces or maintenance sensitive equipment or sections thereof.

After this short list, the focus was directed towards plain metal surface with maximum heat transfer properties to avoid interface reaction between liquid silicon and the solid cooling surface.

3. EXPERIMENTAL WORK UNDERTAKEN WITH A SEMI-STATIONARY COPPER PLATE

Silbec carried out the first tests of copper thin casting in 2001. The experimental device of these starting tests (Figure 1) consisted of a copper plate with internal pipes to allow heat extraction by coolant circulation, manufactured by *FAI Production*, a French company specialized in copper mouldings. The plate itself was mounted on a frame with a horizontal axis whereas the ladle was hanging in its trunion support of the bridge crane. Thus distribution and the height of free fall of the silicon stream could be easily controlled and adjusted. A hydraulic cylinder allowed to tilt the plate over when the silicon had solidified to evaluate whether a free sliding of silicon from the mould would be possible or whether the Si would stick to the copper surface, so that mechanical scraping would be required. It was also evaluated whether at essentially the same thickness of product a correlation would exist between the amount of heat extracted per time unit and the physical properties of the product thus solidified in order to determine the fractioning behaviour of these different product types when dumped from the plate into the receiving bin underneath. A layer of 1 to 2 cm of liquid silicon cast from a ladle was spread on the plate (Figure 2). One of the early results proved that due to the very fast cooling process, the product obtained had the desired brittleness and was easily breakable under the impact of the fall from a relatively minor height.

More than 500 tests have been performed to understand not only the above-mentioned factors, but also to determine the flow behaviour of silicon at various temperatures and thereby viscosities with silicon either freely falling on the static copper bed or simulating vectorial influences of the stream by moving the ladle in predetermined directions and at predetermined speed over the surface of the plate.

The results of all these tests allowed us to determine not only the minimum amount of heat to be extracted as per formulae (1), (2), and (3) below, but also the surface area needed to allow an offering of a clean mould surface at a controlled temperature as long as silicon would pour out of the ladle.

The evaluation of the test results of several months enabled us to determine the parameters for the design of our thin cast installation. These were, among others, the cooling system of the copper plates, the best way of thin casting of the liquid metal, and the stripping technique. All these factors were evaluated and measured with the aim of setting up a mechanically simple installation while being reliable and secure.

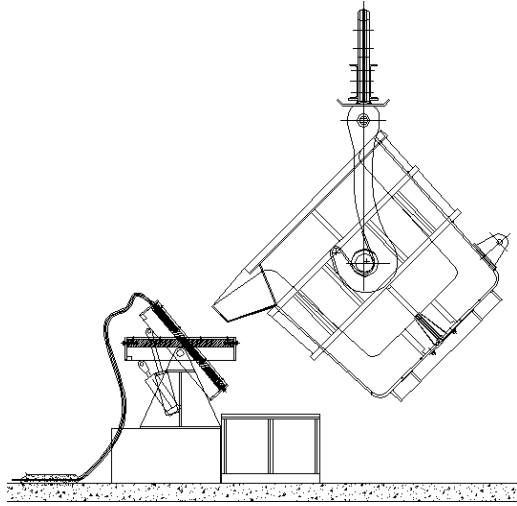


Figure 1. Experimental device for the first cast tests on copper.

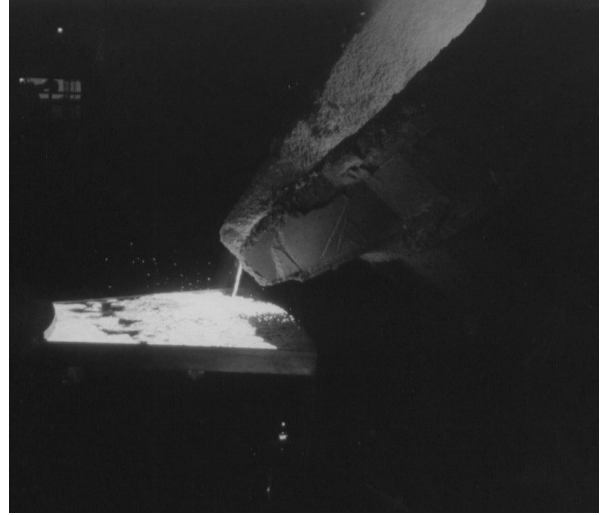


Figure 2. Thin cast test on copper plate.

The theoretical heat extraction requirements for the design of our machine were based on heat transfer calculations with a liquid silicon flow of 4 kg/sec at 1500°C, and stripping at 1000°C. The calorific power having to be absorbed by the copper carousel was calculated as follows:

$$Energy_{1kg} = (1500 - 1412)^{\circ}C \times 0.968 \frac{kJ}{kg \times ^{\circ}C} + 1787 \frac{kJ}{kg} + (1412 - 1000)^{\circ}C \times 0.946 \frac{kJ}{kg \times ^{\circ}C} = 2262 \frac{kJ}{kg} \quad (1)$$

where: $Energy_{1kg}$ is the energy needed to heat 1 kg of silicon from 1000 to 1500°C.

$$Power_{4kg/s} = 4 \frac{kg}{s} \times 2262 \frac{kJ}{kg} = 9.0 MW \quad (2)$$

where: $Power_{4kg/s}$ is the power to be removed from liquid silicon at 1500°C to reduce its temperature at 1000°C, at a casting flow of 4 kg/s inclusive of transfer from liquid to solid.

$$Power_{Cu} = \frac{9.0 MW}{2} = 4.5 MW \quad (3^*)$$

where: $Power_{Cu}$ is the power the cast copper system must be able to remove to prevent heating the copper plates.

These theoretical calculations were reasonably in line with the results obtained from the practical tests on the single plate, so that a dimensional layout determining the total copper surface and the optimal subdivision into individual copper segments – with sizes and dimensions still allowing a commercial manufacture and treatment – could be done still enabling uniform heat extraction through the cooling system.

* Assuming that solidification fronts of the lower and upper surface of the cast silicon meet in the center of the silicon layer.

For this specific task the accumulated knowledge of our development partner *FAI Production* was vital as it allowed us to determine the total copper surface, the minimum copper volume, the optimum arrangement for the cooling channels in the copper plates, the optimum number of subdivisions into identical sections, as well as the choice of the heat extraction medium.

4. LAYOUT AND CONTROL PARAMETERS FOR THE CASTING MACHINE

The following two evaluations had a decisive influence on our layout:

- The heat flow evaluation in the experimental copper plate as shown in Figure 3.
- A surface erosion test of the copper surface to understand the impact of the temperature of the liquid silicon and of the height of a free fall on potential surface damage.

Figure 3 shows the temperature curve of the copper plate of the experimental device during repetitive casting and removing of fresh liquid silicon while operating the cooling device at the optimal parameter. It shows that after 5 minutes a steady state is achieved, and additional repetitions of casting and tilting do not raise the temperature above the level of about 170 to 180°C, and that at the end of this test, after about 20 minutes, a relatively rapid temperature decrease could be achieved so that actually continuous casting can be done if liquid metal would be continuously available.

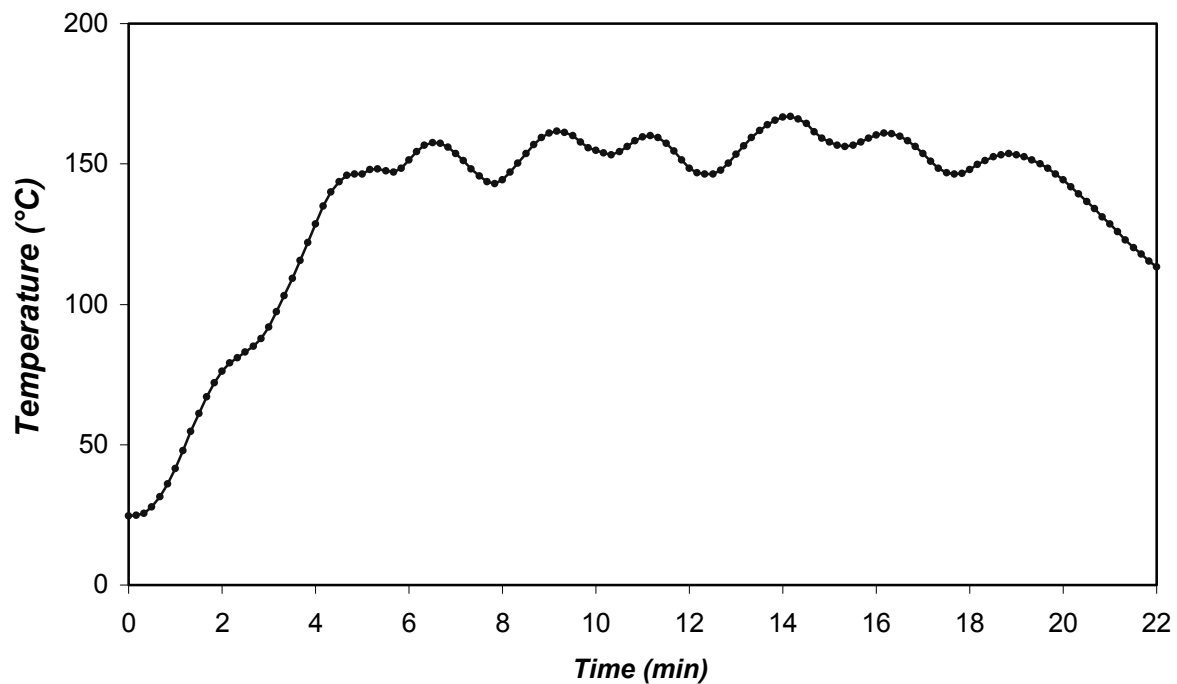


Figure 3. Temperature of the copper plate equipped with the chosen cooling system.

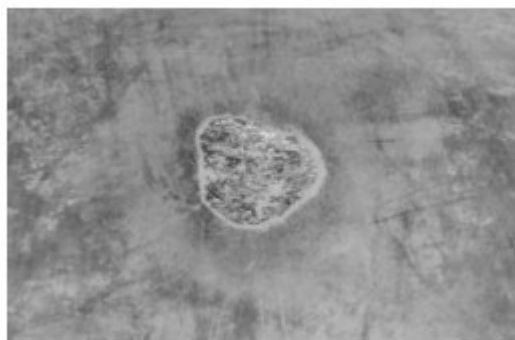


Figure 4. Copper plate erosion through a hot point.

Figure 4 shows the surface erosion caused by the impact of overheated silicon at a temperature of more than 200°C above melting point and an intended relatively high free fall. This formed a cavity of a depth of about 5 mm and an erosion crater of about 70 mm at a given amount of silicon thus freely falling onto the copper surface. This test also allowed to determine the importance of the vectorial movements of the liquid stream to be designed in horizontal direction compared to the undesired monodirectional force of the static free fall with no horizontal design flow components.

5. BRIEF DESCRIPTION OF THE LAYOUT OF THE CASTING MACHINE, AND CONTROL PARAMETERS INCORPORATED INTO IT

The accumulated experience with silicon metal granulated in water for more than eight years without any major damage to our casting equipment because of the extremely precise control devices and mechanisms allowed us to use this very same design of the ladle tilter and to adjust it to the requirement for the copper carousel, so that a steady control of the flow of liquid silicon on the copper plate can be warranted and the length of time between the repetitive fall of liquid silicon onto the very same copper surface would be maximized.

Sixteen identical copper segments form a huge horizontal wheel with an entirely flat surface exposed to liquid silicon. The distribution of the liquid silicon on the surface is determined by the amount of silicon per time unit added to the surface, and the speed of revolution of the wheel. Before one complete rotation of the wheel, appropriate devices remove already solidified silicon so that it falls into a collection bin with a vibrating feeder underneath, from where it is transported into the holding boxes. Heat shields to protect the surrounding equipment both at the centre of the wheel and towards its outer edges in the area where the mould tilter is operated are also vital for minimizing splashing. All operating parameters are controlled and adjusted from an operating cabin at an elevated level close to the casting wheel. There are a series of limit switches and interlocks, so that even operating errors would not bring the casting beyond certain critical limits, and failsafe devices are incorporated.

All mechanically moveable parts are designed so that they are not directly exposed to any potentially liquid silicon. The drive design is so that a minimum of mechanical equipment is used, and any failure would immediately cause the failsafe features to engage. The centre of the wheel containing the inlets and outlets of the cooling media into and from each copper section are effectively shielded, and there are no electric connections in the centre either.

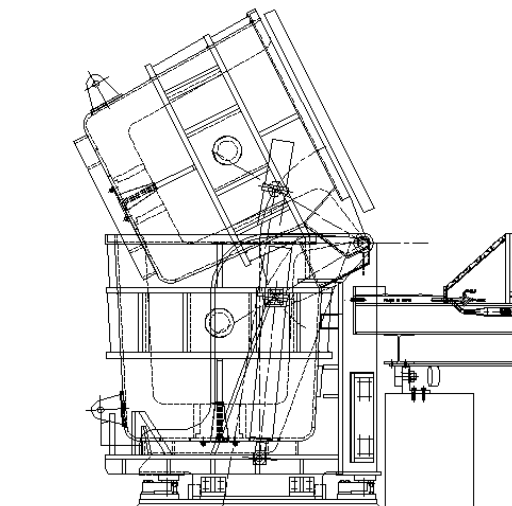


Figure 5. Ladle tilter radially moveable to casting wheel.

6. INITIAL OPERATING EXPERIENCES

In starting up the equipment (Figures 6 and 7), priority was put in observing specific criteria related to workplace safety and security, and protection of critical parts. This included a systematic hot-run test period during which the operations were actually run and controlled by *Silbec's* engineering team.

On-the-job training was then provided so that initially a one-shift operation and subsequently further use of the installation towards the ultimate continuous around-the-clock operation would be achieved.

Obviously, as with every new extra-heavy duty equipment design, certain modifications became necessary, but none of them affected the basic principle. At the end of an eight-hour operation of the machine with occasionally more than the metal from just one furnace no copper temperature beyond 200°C during the entire operation was observed.

During the extended start-up it happened that our biggest furnace – 30 MW – was on a campaign of FeSi 75 and higher Si containing specialties[4][5], so that we were able to test the casting carousel with these products as well. Of particular interest was to observe the fractioning behaviour of certain specialty products produced during this campaign as well as to accumulate products for evaluation and acceptance by our various customers.



Figure 6. Thin cast installation.



Figure 7. Operation of casting onto the copper carousel.

7. EVALUATION OF THE PRODUCT OBTAINED FROM THE CASTING WHEEL

During the initial moments of liquid silicon metal touching the copper surface until the first solid crust formation, heat flow is essentially monodirectional. During solidification, stresses induced by thermal dilatation and crystal growth cause the silicon metal to deform slightly and as a result breaking of the contact between the solidified silicon and the copper base. This is the beginning of the separation of the two surfaces, and from this moment on, heat conduction is substituted by radiation, and consequently the rate of heat extraction slows down.

Even though the silicon on the plate is still red at the surface after 60 seconds, it can be tipped over into the collecting metal box. Solid silicon never sticks to the copper, and after deflecting it from the carousel, the copper plate is free from any material.

Silicon produced by casting in moulds solidifies in about 30 minutes, whereas silicon granulated in water needs just some seconds. On our casting wheel, this process takes about 60 seconds. Such fast cooling results in a very homogenous product that could improve the performance of the product in certain downstream processing. Indeed, the solidification speed directly influences the grain size and the primary impurity distribution of the silicon (= formation and composition of intermetallics)[6]. As impurities have less time to segregate, the uniformity of the chemical distribution of each piece is much higher than traditionally cast silicon.

During the tests a certain amount of very shiny silicon was produced at low casting temperatures, as no surface oxidation had taken place (Figure 8a). When casting at 1550°C and higher, a thin oxide layer of whitish appearance formed at the surface. Its maximum thickness, as measured by the electronic microscope, is about 1µm (Figure 8c). While optically remarkable, the total SiO₂ contamination thus added is less than 100 PPM by weight (g/MT), thus with no negative impact on product utilization whatsoever.

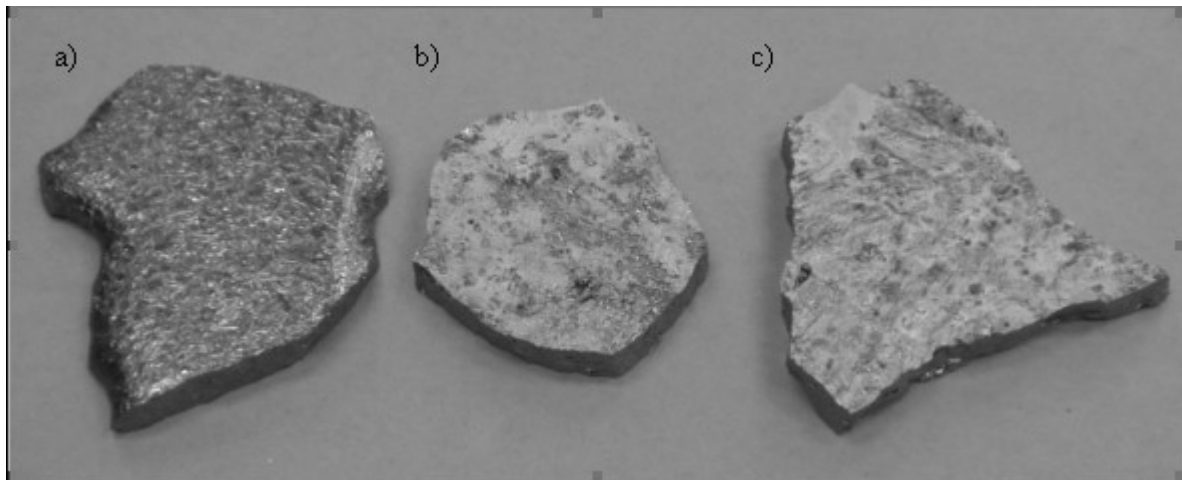


Figure 8. Silicon metal cast on a copper plate.

When casting silicon metal on our machine destined for standard sizes for the aluminum industry, no mechanical crushing whatsoever is required thereafter. In order to properly size the product, the material is fed directly to the multi-deck sizing machine, thus bypassing the existing crushing equipment. Basically, when the product falls off the copper plate into the collecting bin it breaks like a china plate falling on a hard floor, and very little small splinters or powder occur.

Figure 9 shows the grain size distribution of silicon metal of the same chemistry cast conventionally and then passing the precrusher, the jaw crusher and the screening deck, compared to the thin cast metal as described after having passed the screening deck. We have determined the quantity of small sized material below 3 mm to be not even 1%, whereas conventional casting and crushing yields above 5%. A separation of small size material at 10 mm gives an improvement of 10% points, i.e., only 2% of downgrading compared to approximately 12% in the conventional crushing and sizing.

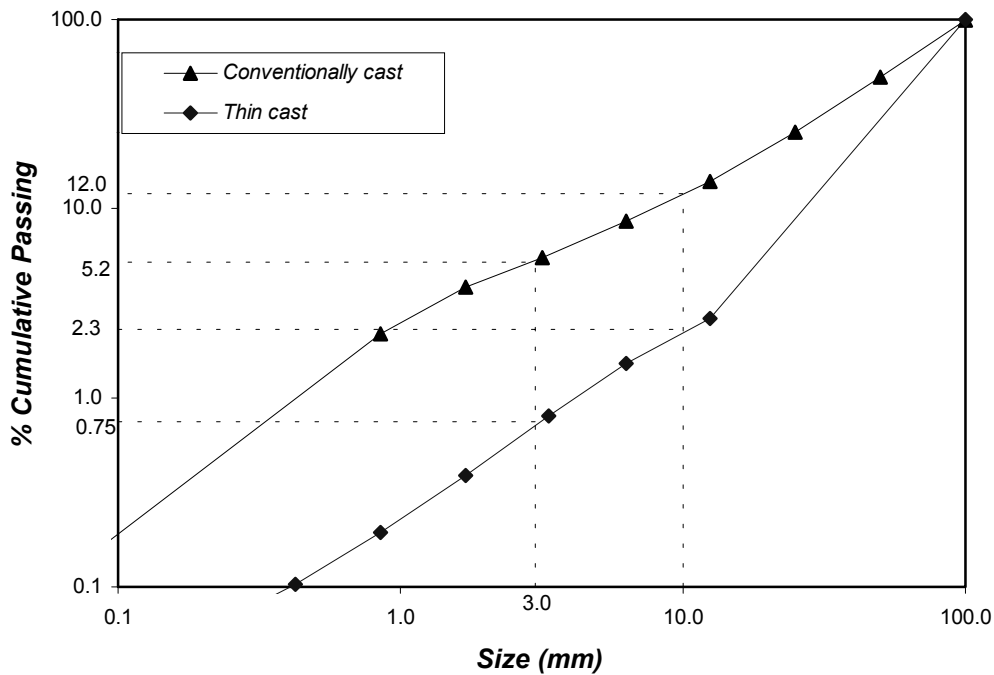


Figure 9. Grain size distribution of conventionally cast, cooled, and crushed silicon to 0 x 100 mm compared to thin cast product

8. CONCLUSIONS AND OUTLOOK

At the time of writing this paper, in the early operation, substantially more than 100 t of silicon were cast over that machine and immediately broken into the desired size. Various small modifications to the machine have become either required or recommendable in order to increase the probability of achieving the previously mentioned postulations. However, none of these changes or additions affected the core of the machine, i.e., size and arrangement of the copper plates, the heat extraction system, the positioning of the ladles relatively to the copper plates, the tilter control, the silicon flow rate, and the flow/distribution vectors of liquid silicon pouring onto the copper plate.

We intend to present further operating results at the conference presentation and, very likely, also report on casting of FeSi 75 and other specialties, which are part of the production program at *Silbec* during certain campaigns.

The initial success of this new equipment justifies once more our trust and confidence in its own technology development efforts, as evidenced by its silicon granulation plant, its flexible electrode technology SKTEC, the injection of small size material through the graphite core of the compound electrodes[7] and the determination of basic factors of alternative furnace technology[8], which became the foundation of all our production process related developments. These various technological developments are *Silbec's* response to the ever-increasing challenge in developed countries to maintain production facilities competitive despite ever enlarging requirements for environmental protection as well as labour, and health & safety costs, unknown to our competitors in developing countries or non-market economies. We firmly believe that strategic Si metal consumers will honour such commitments of a producer by awarding him at least the preferred supplier status.

9. REFERENCES

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