# Development and Testing of Super Reinforced Ceramics

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**Abstract**

*Super Reinforced Ceramic is a load bearing refractory composite utilising a complex multicomponent ceramic mixture. It is mainly used in the metal and mineral processing industries where the material must stand up to high temperatures and high loads. This project looks at developing the composite to improve its strength and manufacturability so that it can be more competitive in the market. Variations of the composite have been made by using alternative aggregates and binders, and testing has shown improvements in compressive and bending strength.*

## 1. Introduction

Super Reinforced Ceramic (SRC) is a steel fibre reinforced composite designed for high temperature, high stress application. Brought to market by Sila Australia in the last decade, the material has been developed based off years of industry experience. The main application of SRC is in the metals and minerals processing industries. SRC has not gone through any formal optimisation process, hence the purpose of this project is to improve both the material properties of SRC, and the ease of manufacturing of the product through testing. Sila is a small, growing company and is currently leading the Australian market in this technology. Competition is expected soon, so Sila must continue to develop their product in order to increase SRC's reliability and effectiveness.

SRC is not a typical refractory material as it is used to make components which must withstand high temperatures, thermal shock, abrasion and mechanical stress via compression, bending and impact. Sila has multiple compositions of SRC to suit different applications; typical applications include dross skimmer blades for molten aluminium or iron processing, air tubes inside rotary kilns and nose ring blocks for the hot end of cement kilns. In certain applications temperatures can reach as high as 1600°C, however typical applications are in the range of 600°C - 1200°C.

Testing at elevated temperatures is not currently feasible due to lack of equipment and cost of external testing, so it was decided that ambient temperature testing of Cold Compressive Strength (CCS) on the ceramic and Modulus of Rupture (MOR), also known as 3-point bending, on the composite would be used to track material improvements.

SRC relies mostly on hydraulic bonding from a cement component to form its strength. However, adding more cement to increase strength is not an option as the cement tends to form liquid phases at high temperatures (McGowan, 2008). So, the aim of the experimentation is to improve the strength of the ceramic component by altering the aggregates and fillers used in the mixture, while not increasing the amount of hydraulic binders used.

# 2. Process

## 2.1 Review of Testing Standards

In order to gather data which is statistically significant and consistent with established testing methods, a review of ceramic and refractory standards was conducted and used to guide development of the testing. Australian Standards for Refractories were used for casting, drying, firing, as well as CCS and MOR testing. ASTM standards were used to develop a procedure for measuring the liquid ceramic (wet mix) consistency while casting.

## 2.2 Development of Moulds

The mould design needs to be optimised in order to allow a large throughput of specimen production, while also maintaining quality and consistency. The MOR moulds were constructed from steel plate and were made to the standard refractory brick size of 230 x 114 x 76 mm as per Australian Standards AS 1774.3—2000.

The moulds for CCS were developed through an iterative process. Initially, steel moulds were used, but it was found that the down time required to prepare and strip these was too long and would hinder production of test specimens. Disposable polystyrene moulds were then trialled, but were found to be too porous which often resulted in voids being left in the samples, and hence, large variations during strength testing. A design using cylindrical 50 mm PVC pipe was settled on, which left the samples with a smooth surface finish and few deformities. As a result, variation in strength has reduced. The three iterations of CCS moulds are pictured in Figure 1.


**Figure 1 Iterations of mould design left (initial) to right (final): Cube shaped steel mould, Polystyrene cylindrical mould, PVC cylindrical mould. Notice the large improvement in surface finish of the PVC moulds as compared to the others.**

## 2.3 Suspension Agents

In one composition of SRC, the fibre is suspended in the liquid ceramic before casting. It is important that the fibres are suspended uniformly because clumping of the fibres, or allowing the fibres to sink to the bottom of the mould will create local reduction of tensile strength. This requires that the liquid ceramic has a suspension agent added to it in order to increase the mixture viscosity.

Two suspension agents, labelled SA1 and SA2 have been compared by casting bricks using varying amounts of the two agents. The liquid ceramic filled moulds are allowed to vibrate on a vibration casting table for a set period of time before the SRC is allowed to cure. After curing, the bricks were cut open and the distribution of fibre in the bricks was inspected visually to identify the effectiveness of the Suspension agents.

## 2.4 Testing Compressive and Bending Strength

Strength testing of the SRC has been done in house at Sila using a 550KN hydraulic press. Material strength testing tends to exhibit significant variation between samples (Hawkes, 1970) so in order to collect data with statistical significance, it was decided to use 2 batches of 15 samples for CCS testing and 3 batches of 4 samples for MOR testing for each mixture tested.

To date, 3 mixtures have been tested. Mixture 1 is the standard ceramic mixture which Sila have been using. Mixture 2 replaces the standard aggregate with an alternative material and Mixture 3 replaces the standard cement binder with a less expensive and more readily available alternative.

SRC properties do not scale down well so CCS of the full composite would require much larger equipment than is available at Sila, so the CCS samples are made using only the ceramic component of SRC. Each sample that is produced has its pressing faces sanded flat and parallel. This is done to reduce stress variations in the sample caused by induced bending moments due to raised parts of the pressing faces. The samples are left to cure for at least four days and are then fired to a standard temperature overnight to drive the excess water from the sample. Finally, each sample is numbered and the height, diameter and weight is recorded. The sample is then crushed as per Australian Standards using a constant force application rate and the maximum force is then recorded and converted to a strength reading.

A similar process is used to produce and test the MOR bricks, however the MOR samples are made with fibre so that the whole SRC composite is tested. The MOR samples require at least 7 days of curing before they can be fired safely. The same hydraulic press is used for testing the MOR samples as is used for the CCS tests. However, a 3-point bending jig is placed in the press to accommodate the bending tests.

## 2.5 Mixture Constituent Ratio Adjustment

Ratios of the SRC ceramic mixture's components are to be adjusted to optimise the liquid ceramic fluidity and setting time. These properties can be adjusted using fillers and additives in the liquid ceramic mixture. Increasing fluidity makes the casting process quicker and reduces the amount of air bubbles trapped in the part. By adjusting the setting time, the liquid ceramic can be made such that there is enough time to perform casting, but will set soon after. This again allows the manufacturing process to be sped up. This work has not yet been completed.

# 3. Results and Discussion

## 3.1 Suspension Agents

Five SRC brick specimens were created for each suspension agent, SA1 and SA2, in quantities varying from 0.2% to 0.7% by mass. The bricks were vibration cast in the vertical orientation, allowing the fibres to sink to the bottom by influence of gravity. After the SRC had cured the bricks were cut open and visually inspected as depicted in Figure 2.

It was evident that suspension agent 2 was not effective for the concentrations used, this can be seen inside the boxed areas where the fibre is lacking. The lack of fibre in these areas reduces the tensile strength locally. This may make the SRC prone to chipping or cracking. Fixing this by increasing the amount of the agent used is not desirable because the agent does not significantly contribute to material strength, and increases the water requirement.

The samples using SA1 showed little-to-no fibre settling at concentrations of 0.45% - 0.575%. However, there is an anomaly at the 0.7% concentration, where significant fibre separation has occurred near the top surface. This was likely caused by the order in which casting occurred, since the 0.7% sample was left vibrating for the most amount of time which likely caused the excess fibre separation.

These results suggest that suspension agent 1 is the most suitable agent to use in the SRC application. A concentration range of 0.45% - 0.575% by mass is required to hold the fibres in place during the casting process.

It was also noticed that voids tend to form in the SRC during casting from trapped air bubbles, seen in figure 2. This is likely to contribute to variation in material strength as seen in section 3.4 and future work will have to explore casting methods to limit formation of these voids.

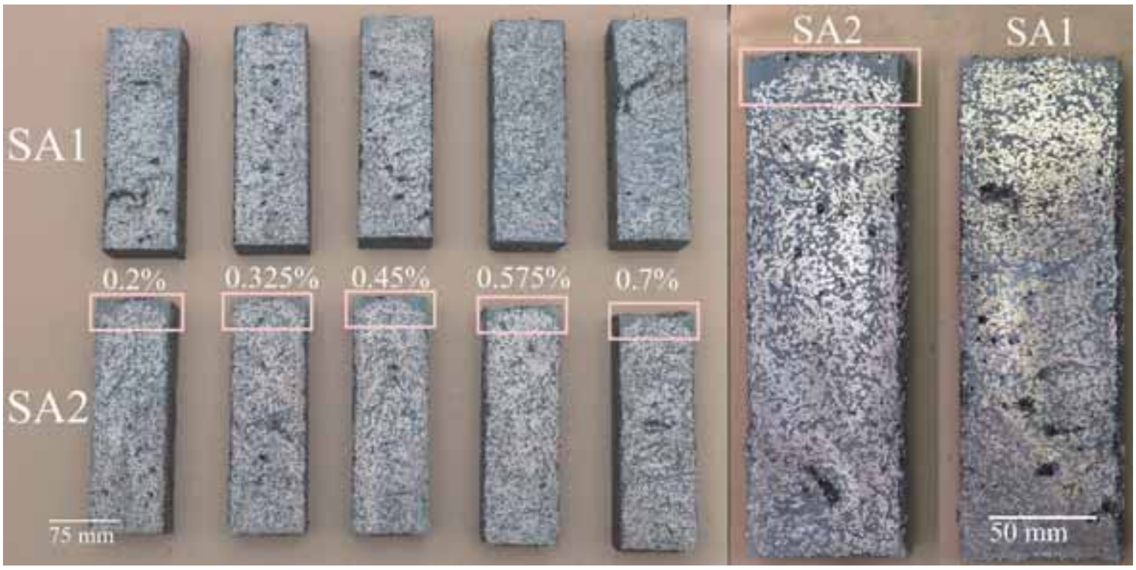


**Figure 2 Fibre distribution in brick specimens using suspension agents SA1 and SA2 in amounts varying from 0.2% to 0.7% by mass. The close up on the left shows the difference of fibre sinkage in detail for the concentration of 0.45%. SA2 seems to be ineffective in all cases as seen by the large regions that are missing fibre outlined in the rectangles.**

## 3.2 Cold Crushing Strength

Three SRC ceramic mixtures were tested for cold crushing strength by testing 2 batches with 15 samples in each batch with the exception of Mixture 1 where 3 batches of 15 were tested. Mixture 1 is the standard SRC mixture, Mixture 2 uses an alternative aggregate to Mixture 1 and Mixture 3 uses an alternative binder to mixture 1. The results are summarised in Figure 3, which show a 45% and 51% improvement in Mixture 2 and Mixture 3 respectively, as compared to Mixture 1.

From the data gathered so far, it is apparent that the two alternative constituents have improved the overall compressive strength for the SRC ceramic. It is surprising that these two alternate materials cause such a significant improvement over the original, and it may be the case that some flaw in the preparation or testing methods has caused premature failure of the earlier specimens.

## 3.3 Modulus of Rupture

The same mixtures, as detailed in section 3.2, were tested for bending strength through MOR. Twelve specimens were created for each mixture and the maximum MOR strength for each was recorded as shown in Figure 3, which shows a 7% and 15% improvement in Mixture 2 and Mixture 3 respectively, as compared to Mixture 1.

Bending strength is dependent on the composites ability to effectively transfer stress into the reinforcing fibres (Termonia, 1990). It is no surprise that Mixture 3 with the alternate binder had a larger change in strength than Mixture 2 with the alternate aggregate, since the binder is what provides adhesion between the ceramic matrix and the fibres.

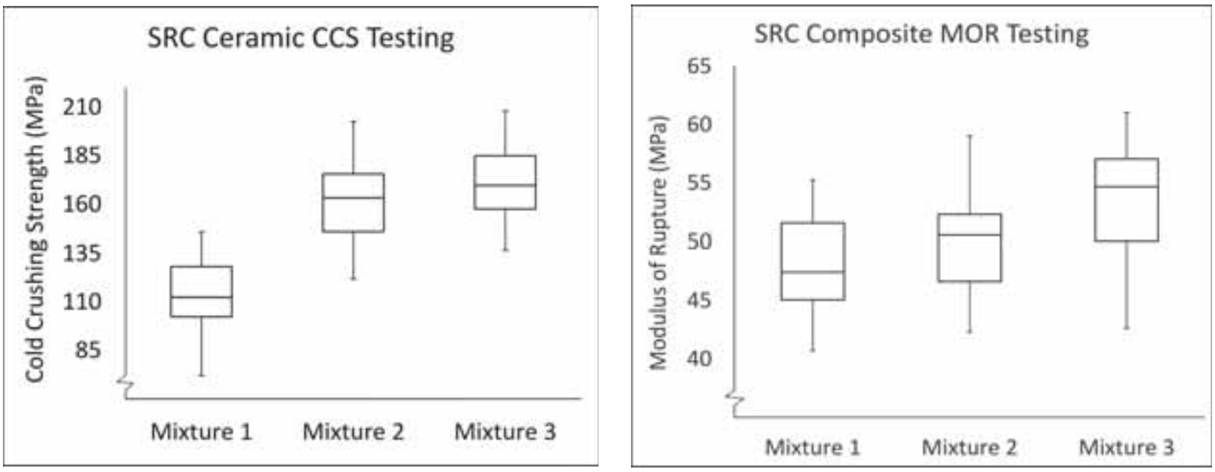


**Figure 3 Cold Crushing Strength and Modulus of Rupture test results. For CCS, Mixture 2 and 3 had a sample size of 30 and Mixture 1 had a sample size of 45. For MOR, all mixtures had a sample size of 12.**

# 4. Conclusions and Future Work

Through testing at Sila’s on-site laboratory, Super Reinforced Ceramic has had compressive and bending strength quantified. Modifications have been made to the mixture and improvements have been tracked, showing significant increases in strength. It is assumed that these improvements will extend the usable product life and reduce maintenance costs and plant shut down time, both of which are desired by Sila's customers.

The remainder of this CEED project will focus on improving the manufacturability of the product, which includes optimising the flow and setting properties for casting. The material strength improvement found in section 3.2 was larger than expected, so will be revisited.

Sila intends to continue their R&D program to gain a deeper understanding of SRC’s properties which will include further strength testing on modified mixtures and measuring stress – strain curves of SRC. In the future, SRC should be tested for three body abrasion and impact loading, as it must withstand these conditions in service. The ceramic’s material properties are expected to change significantly as the hydraulic bonds begin to break down at high temperatures (Strauss Rambo, 2016), so strength and abrasion testing should also be conducted at elevated temperatures to gain a better understanding of the material behaviour. Additionally, the matrix – fibre interface will be studied to understand how stress is distributed through the material.

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