

**NPL REPORT
DEPC MPE 026**

Studio Project: DISIC

**Dimensional stability of
ceramic casting moulds**

**R Morrell, P N Qusted, S Jones
and D A Ford**

UNRESTRICTED

April 2006



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NPL Report DEPC MPE 026

Date: April 2006

National Physical Laboratory**Studio Project DISIC –
Dimensional stability of ceramic casting moulds****by****R Morrell, P N Qusted, S Jones* and D A Ford******Division of Engineering and Process Control*****University of Birmingham, IRC in Materials Processing****** DAF Associates****Summary:**

This report summarises the results of the NPL Studio project DISIC in which the dimensional stability of ceramic shell investment casting moulds has been investigated using a suite of laboratory tests as well as plant casting trials. Shell moulds of 'generic' types were manufactured by the University of Birmingham, and industrially employed mould types were supplied by four industrial partners. Different behaviours of different types of shell systems have been identified. The full silica system has been shown to be the most dimensionally stable except in a low temperature regime between 800 °C and 1000 °C during initial firing. A full aluminosilicate system was the least dimensionally stable at higher temperatures during casting. Zircon/molochite and zircon alumina systems have intermediate behaviour. The dimensional deviation obtained from the initial wax shape to the cast metal shape was investigated using a plate casting test. The results of these trials broadly paralleled the laboratory tests.

The complex temperature and stress distributions during casting cannot be directly modelled in laboratory tests, and success in casting is related also to the size and shape of the casting, as well as the thickness of the mould created around the wax precursor. Nevertheless it is believed that the results of the project enable users to rank the shell systems according to risk of deformation and dimension change during the various stages of pre-firing and casting.

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NOTE:

This document reports the detailed results of a collaborative study of the DTI ‘Studio’ type undertaken at NPL and the University of Birmingham with input from a number of organisations involved in high-temperature alloy investment casting. The principal driver was to improve understanding of the role of the mould material in achieving dimensional stability during casting. The project was established to provide a baseline for comparing the performance of different types of mould system and correlating it with material properties and high-temperature deformation characteristics.

The initial reporting of the project was to the DISIC industrial consortium only. Under the terms of the Studio contract, the results remained confidential to the consortium for a period of one year, after which they were to be placed in the public domain, although the identification of the collaborating partners mould systems remains confidential.

1. INTRODUCTION

The dimensional stability of ceramic investment casting moulds through their manufacturing process and the process of casting the metal shape is critical for obtaining the correct dimensions of the cast shape. Size and/or shape allowances have to be made for mould distortions, which result in oversize castings and hence additional machining costs to obtain the required dimensions and tolerances. Following the NPL Studio Project 'PAMRIC' (1) in which some alloy and mould property data were gathered, this present Studio Project entitled 'Dimensional Stability of Investment Casting Moulds' (DISIC) was conceived to understand better the dimensional changes that occur in moulds in each stage of their manufacture and use.

Ceramic investment casting moulds are made by a multistage coating process applied to a wax former of the required shape (see Figure 1), the thickness of several millimetres being built up by successive layers applied via slurry dipping or spraying with a fine suspension of bonding material, with coarse particles being applied either by a 'rain' technique or by fluidised bed. After drying the bonding material, the wax is carefully removed, typically by a steam autoclave process ('de-waxing') which is controlled to minimise the risk of the wax thermal expansion bursting the mould.

There is a wide range of material possibilities for the construction of an investment casting mould:

1. The metal contact face is usually a fine grained refractory material, typically alumina or zircon, which can present a smooth face to the metal; this is known as the 'primary' coat;
2. The first backing layers, known as the 'secondary' coats, comprise a bonding material which, in modern aqueous based production routes, is normally a gelling system based on silica sol or hydrolysed ethyl silicate, reinforced with a fine 'stucco' or refractory grain material which may comprise fused silica, molochite (fired clay), mullite, zircon or alumina.
3. The subsequent 'secondary' backing layers are usually similar to the first backing layers in bond type, but may have coarser 'stucco' material, up to 1 mm particle size.
4. A seal coat may optionally be applied, which has the function of bonding the outermost stucco.

The mould thickness may vary considerably depending on the perceived requirements for strength or resistance to distortion during casting, and these are in turn dependent on the metallostatic pressure head which is related to the size of the mould. Typically, large flat areas in the moulded shapes require greater thicknesses of casting mould than smaller rod-shaped features. Sharp corners on the moulded part tend to result in thinner mould build-up, and are often a limiting factor.

Since casting moulds are made as green or only partially fired ceramic bodies which possess only sufficient strength for handling and thermal shock resistance to withstand the casting process, and which change during the casting process as a result of pre-heating and then casting molten metal, there are a number of stages from the point of manufacture to use (Figure 1) in which unwanted deformations can occur:

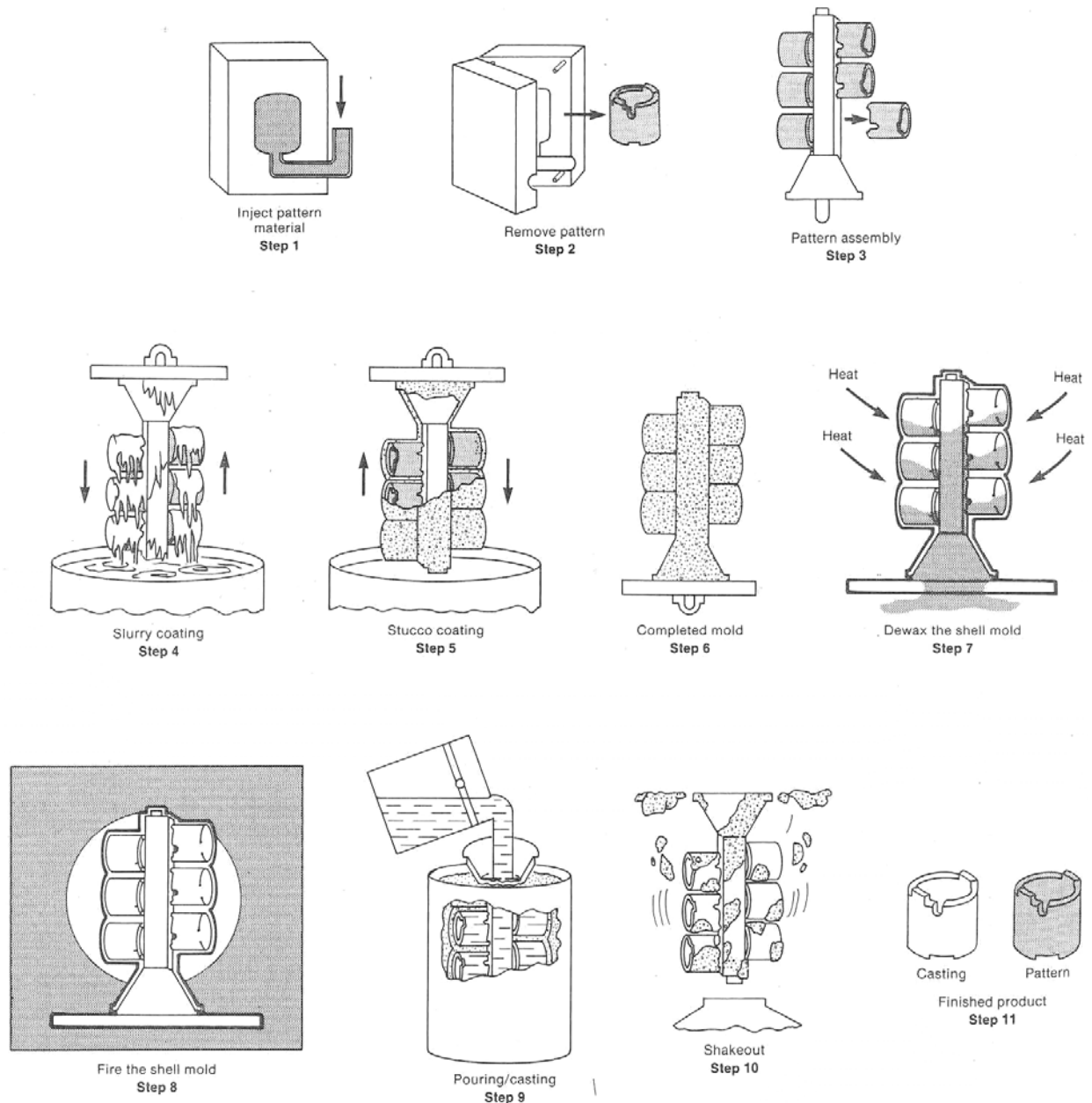


Figure 1: Steps in the manufacture and use of investment casting moulds (from (2)).

1. Wax former deformation can result from creep at ambient temperature, especially in complex shapes and runner systems;
2. De-waxing can exert expansile pressure on the mould, and lead to cracking. Great care has to be taken to minimise the risk of mould fracture¹;
3. After de-waxing and before use, moulds are pre-heated to a temperature typically in the range 850 °C to 1100 °C. This process removes residual wax and combined water from the bonding medium and forms a glassy or ceramic bond. This can be accompanied by a natural sintering size change, and there is an added risk of distortion under gravity during this stage;

¹ Recent work at the IRC Birmingham has been to investigate the behaviour of the mould during de-waxing (3).

4. During the casting process, the pre-heated² mould is subjected to rapid heating by the molten metal. The metal contact face clearly becomes hotter than the body of the mould, but the overall potential for creep is determined by how hot the whole mould body becomes, and this is related to the thermal mass of the mould relative to the thermal mass and latent heat of fusion of the metal adjacent to it, as well as the extent to which heat loss from the mould is controlled by externally applied thermal insulation. The magnitude of mould dimensional change during this process is determined by the pressure exerted by the molten metal, the duration before the metal solidifies and shrinks away from the mould wall, the temperature distribution achieved in the mould, the sintering shrinkage of the mould and its thermal expansion behaviour.

This complex behaviour is difficult to model. Most casting organisation use experience and empirical rules for selecting mould materials and making appropriate allowances for distortion. This Project set out to compare the propensity for different mould materials to distort when tested in equivalent ways, and thereby provide a ranking tool which permits discussion on options for improving industrial practice.

2. THE 'DISIC' STUDIO PROJECT

2.1 Project partnership and project meetings

DISIC was established as a 'Studio' project supported by the DTI Materials Metrology 'Performance' programme. A 'Studio' project is one in which there is at least 50% of the total costs are direct industrial involvement, and 50% can be supported by DTI. Thus typically, industry is expected to inject about 15% in direct funding, and 35% in-kind funding via a variety of routes including material supply, consultancy, or experimentation.

Table 1: Project partnership

Organisation	Role
National Physical Laboratory	Project management, mould testing
University of Birmingham, Department of Engineering	Subcontractor, generic mould material manufacture, casting trial evaluation
DAF Associates	Project chairman, industrial liaison
Rolls-Royce plc	Partner, metal casting trials, establishing testing conditions
Remet Ltd	Partner, mould precursor materials, technical input and feedback
Minco Ltd	Partner, mould materials, particle size data, detailed mould properties
Precicast SA	Partner, mould materials, technical input and feedback
PCB	Partner, mould materials, technical input and feedback
Zollern GmbH	Partner, mould materials, technical input and feedback

² Some metal casting organisations cool the mould and store it before re-heating and casting. The development of cristobalite during the pre-heating is clearly a factor in controlling the properties of the mould during casting.

In the present project, the partnership was developed by DAF Associates, and the members and their roles are shown in Table 1. Additional support and hosting of meetings was provided by the Cast Metal Federation. The project commenced in October 2003, and most of the experimental work was conducted during 2004. Project meetings were held on the following dates:

20 October 2003:	Project set-up – decisions on mould types
28 April 2004	Preliminary results reporting
17 November 2004	Results reporting and discussion

2.2 Project rationale

In order for this project to achieve its objectives whilst preserving industrial confidentiality, it was agreed that there should be a mixture of generic and commercial mould materials. The generic materials would have fully disclosed compositions and structures, and would be made by Dr S Jones of the University of Birmingham. Each partner would have the opportunity to submit their own proprietary mould material, the nature and origin of which would remain undisclosed to the other partners. In this way each Partner could see how his own mould compared with others and with the generic systems without breach of confidentiality.

It was recognised from the outset that most of the test methods required regular geometry test-pieces to be cut from the moulds, and that particularly with regard to the mechanical testing it was necessary to flatten the external stucco surface by flat-bed grinding to create a parallel faced test-piece. Moulds were therefore to be supplied in a form suitable for testing, preferably as flat strips or plates.

2.3 Testing rationale

The principal purpose of the project was to identify the relative deformation behaviour of a variety of mould systems, and to do this required a series of separate measurements to segregate all the possible contributing effects. The following issues were identified:

- Natural shrinkage as a result of sintering during pre-firing and casting;
- Thermal expansion behaviour, to determine the hot mould dimensions relative to starting dimensions;
- The bowing of moulds due to internal pressure from molten metals when filled;
- The compaction of moulds due to contraction of solidifying and cooling metal inside.

There was considerable discussion at the outset of the project concerning the thermal treatment that the mould materials could be exposed to in laboratory tests compared with the conditions seen in real use. By consensus, it was agreed that all mould samples would be subjected to a pre-firing of 1000 °C for 2 h before raising the temperature to the required maximum to simulate the casting operation. Because the moulds contain some silica, and therefore would develop cristobalite during heating, it was important not to undertake the pre-firing as a separate operation to the testing, but to incorporate the pre-firing in the measurement. Heating rates as fast as possible would be used, although clearly they would be limited by the apparatus capability. The testing duration at the peak temperature would be short, typically no more than 4 minutes, to simulate the short time that the mould would remain hot with molten metal inside it. The stresses to be applied in mechanical testing were estimated to be equivalent to the stress generated in a mould wall as a result of a metalostatic head of 1 m for a typical nickel alloy, which generates a bursting pressure of about 0.075 MPa

(10.6 psi). In practice, of course, the mould wall stress is a combination of thermal stress from the temperature gradient to which it is subjected, gravitational stress from its own mass, and pressure-induced stress, which is clearly related to geometry and wall thickness. It was agreed that most of the tests should be performed under a worst-case scenario of 0.5 MPa (72.5 psi) tensile or compressive stress within the test-piece. Annex 1 provides a mathematical rationale for stress level selection.

The project partnership also decided to evaluate two other characterisation techniques. One was the measurement of mould permeability as embodied in BS 1902 Part 10.2 (4), to be undertaken by University of Birmingham. The other was a casting test in which a mould for a plate-shaped casting is subjected to real casting conditions. The difference in shape between the carefully dimensioned precursor wax and the casting would be determined by mechanical measurement, particularly the distortion of the flat faces. This test was originally proposed in work performed by the National Engineering Laboratory in the early 1990s (5). In the present project the University of Birmingham made the plate moulds, and the metal casting was undertaken by Rolls-Royce. UoB also supplied waxes to four of the partners for preparation of their own moulds using their proprietary methods, followed by casting trials.

The University of Birmingham also undertook some baseline tests at room temperature on the mould materials, specifically, room temperature flexural strength (modulus of rupture, MOR) and edge strength³, each in the wet, dried and fired condition.

2.4 Generic moulds

Significant discussion occurred during the early phases of the work concerning the generic moulds. It was agreed that the systems listed as 1 to 6 in Table 2 would be employed, to be representative of the range of systems employed for high-temperature casting of, principally, nickel-based superalloys.

In practice, system 6 was not made, but three systems were repeated in the light of experience with the first five, which produced moulds that were thinner than expected, with some of the mould structures showing insufficient strength to withstand the bulge test casting trials. Thus, mould types 1, 2 and 3 were repeated but with additional secondary layers to increase the thickness.

The silica mould system used a silica-filled primary, while the remainder used a zircon filler.

2.5 Partner moulds

Partners were invited to prepare and submit moulds for testing, and of these, Minco, Zollern, PCB and Precicast provided flat plate and/or strip mould samples prepared on metal or wax formers, in sufficient quantity to permit the variety of tests to be undertaken. In this report, these moulds are referred to as Partner 1, 2, 3, and 4, without the identity of the supplier or the nature of the mould being disclosed.

³ The edge strength test has been developed by the University of Birmingham as a measure of the mechanical strength of thin mould regions developed around sharp corners in the cast shape (see 4.1.2). A standardised V-shaped specimen cut from the around the moulded edge is subjected to a compression test in which a wedge is pushed into the test-piece on the wax side. The force required to fracture the edge is related to its thickness at the tip of the edge, and is converted to a stress in MPa.

Table 2: Agreed generic mould constructions

Generic system	Description	Coat	Slurry*	Stucco**	Air Speed (ms ⁻¹)	Dry Time (hours)
1	Full Silica 1	Primary 1	NPL/2003/01	Min-Sil 50/100	0.4	24
		Secondaries 2 - 3	NPL/2003/03	Min-Sil 50/100	3	1.5
		Secondary 4 - 7	NPL/2003/03	Min-Sil 30/50	3	1.5
		Seal coat 8	NPL/2003/03	n/a	3	24
2	Full Alumino-Silicate 1	Primary 1	NPL/2003/02	Molochite 50/80	0.4	24
		Secondaries 2 - 3	NPL/2003/04	Molochite 30/80	3	1.5
		Secondary 4 - 7	NPL/2003/04	Molochite 16/30	3	1.5
		Seal coat 8	NPL/2003/04	n/a	3	24
3	Zircon/Molochite 1	Primary 1	NPL/2003/02	Molochite 50/80	0.4	24
		Secondaries 2 - 3	NPL/2003/05	Molochite 30/80	3	1.5
		Secondary 4 - 7	NPL/2003/05	Molochite 16/30	3	1.5
		Seal coat 8	NPL/2003/05	n/a	3	24
4	Zircon/Alumina 1	Primary 1	NPL/2003/02	Fused Alumina AL80	0.4	24
		Secondaries 2 - 7	NPL/2003/05	Tab Alumina 28/48	3	1.5
		Seal coat 8	NPL/2003/05	n/a	3	24
5	Zircon/Alumina 2	Primary 1	NPL/2003/02	Fused Alumina AL80	0.4	24
		Secondaries 2 - 7	NPL/2003/05	Tab Alumina 14/28	3	1.5
		Seal coat 8	NPL/2003/05	n/a	3	24
6***	Full Alumina	Primary 1	NPL/2003/07	Fused Alumina AL80	0.4	24
		Secondaries 2 - 3	NPL/2003/06	Tab Alumina 28/48	3	1.5
		Secondary 4 - 7	NPL/2003/06	Tab Alumina 14/28	3	1.5
		Seal coat 8	NPL/2003/06	n/a	3	24
7	Full Silica 2	Primary 1	NPL/2003/01	Min-Sil 50/100	0.4	24
		Secondaries 2 - 3	NPL/2003/03	Min-Sil 50/100	3	1.5
		Secondary 4 - 9	NPL/2003/03	Min-Sil 30/50	3	1.5
		Seal coat 10	NPL/2003/03	n/a	3	24
8	Full Alumino-Silicate 2	Primary 1	NPL/2003/02	Molochite 50/80	0.4	24
		Secondaries 2 - 3	NPL/2003/06	Molochite 30/80	3	1.5
		Secondary 4 - 9	NPL/2003/06	Molochite 16/30	3	1.5
		Seal coat 10	NPL/2003/06	n/a	3	24
9	Zircon/Molochite 2	Primary 1	NPL/2003/02	Molochite 50/80	0.4	24
		Secondaries 2 - 3	NPL/2003/07	Molochite 30/80	3	1.5
		Secondary 4 - 9	NPL/2003/07	Molochite 16/30	3	1.5
		Seal coat 10	NPL/2003/07	n/a	3	24

* See Tables 4 and 5

** See Table 3

*** Not manufactured

3. MOULD MANUFACTURE

3.1 Raw materials

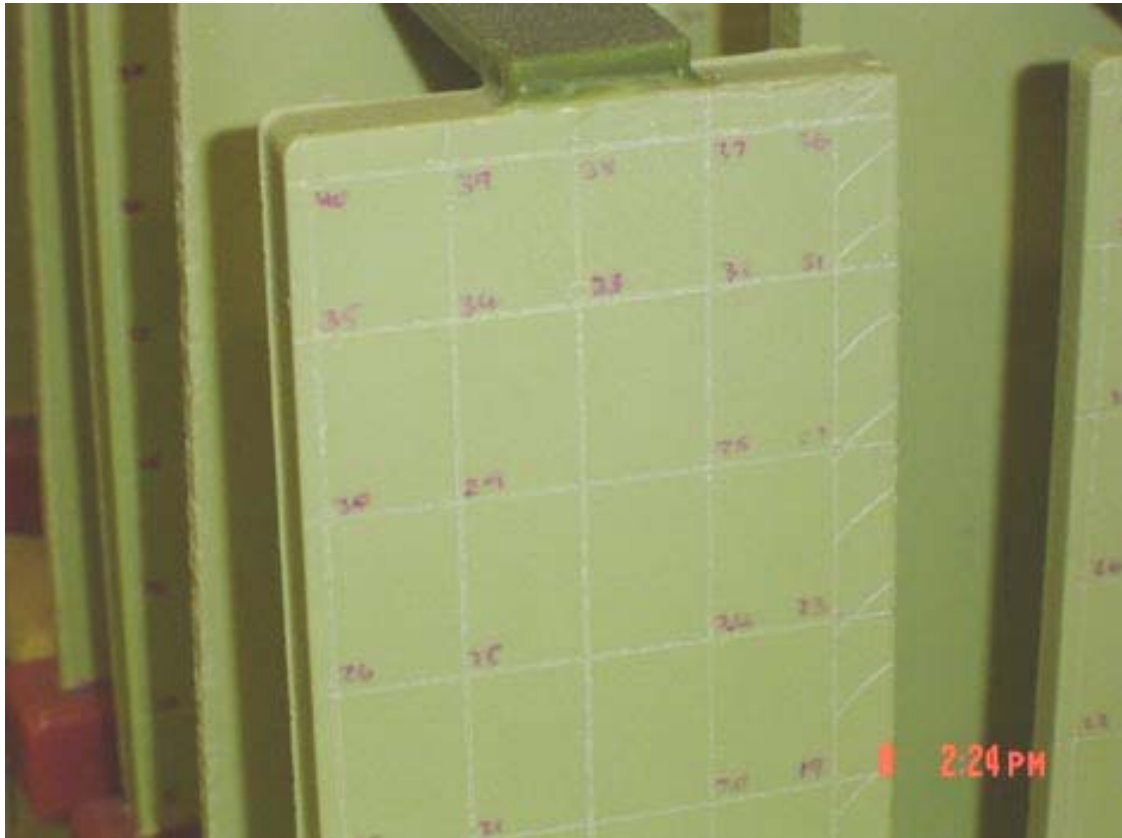
Table 3 lists the principal ceramic raw materials employed for mould manufacture at the University of Birmingham. A water-based, latex-modified colloidal silica binder system, Remasol LP (Remet), was used as a primary binder, and Remasol SP (Remet) was used as the secondary binder. The latex modification was Remet Ultra, which is the latest version available for water-based slurries. All systems were prepared using Victawet 12 as the wetting agent and Burst RSD10 as the antifoam agent. See Annex 2 for granulometry details.

Table 3: Raw materials for manufacture of moulds

Raw material identification	Date received	Material description	Supplier	Supplier reference
Studio 1	05-Dec-03	Min-Sil 120F	Minco	325013 SPS-015
Studio 2	05-Dec-03	Min-Sil –50/+100	Minco	304616 SPS-012
Studio 3	10-Dec-03	Min-Sil –30/+50	Minco	325210 SPS-009
Studio 4	06-Dec-03	Tab alumina 14/28	Precicast SA	2818.1
Studio 5	15-Dec-03	Fused white alumina 200 mesh	Remet	Batch 21727
Studio 6	12-Jan-04	Zircosil 200M	Johnson Matthey	Delivered Sept 2001
Studio 7	12-Jan-04	200 Molochite (<0.075 mm)	ECC International	
Studio 8	12-Jan-04	50/80DD Molochite (0.18-0.30 mm)	ECC International	
Studio 9	12-Jan-04	30/80DD Molochite (0.18-0.50 mm)	ECC International	
Studio 10	12-Jan-04	16/30DD Molochite (0.5-1.0 mm)	ECC International	
Studio 11	12-Jan-04	AL80 (80 mesh brown fused alumina)	Universal Abrasives (Rolls-Royce)	AAS 80
Studio 12	12-Jan-04	AL +60 (28/48 mesh tabular alumina)	Universal Abrasives (Rolls-Royce)	ATR 3285
Studio 13	12-Jan-04	Calcined zircon sand, 80 mesh	Johnson Matthey	

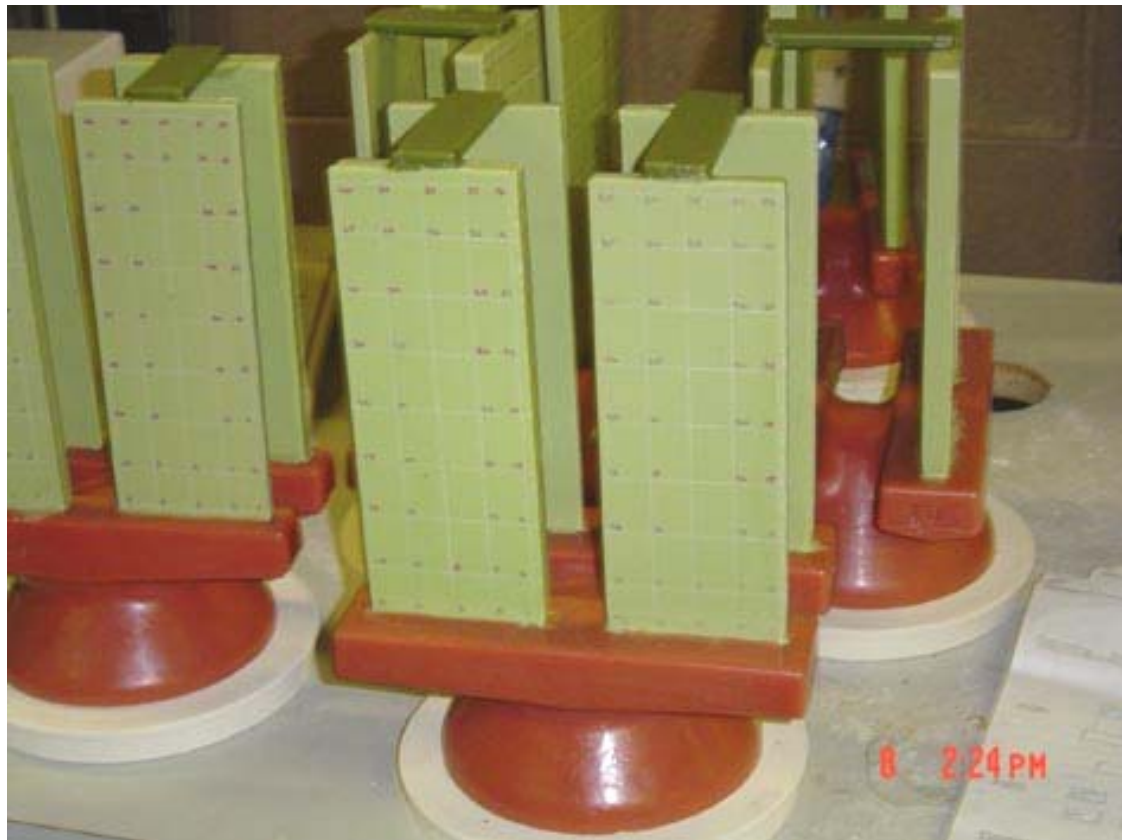
3.2 Wax preparation

Waxes were prepared by Rolls Royce CRDF Bristol for the University of Birmingham using standard size steel moulds on a Muller Phipps injection machine. The wax formulations were: Green Pattern Wax 1: KC-3011-N; Red Runner Wax 1: KC-2690-F; Green Runner Wax 2: Remet Hyfill. For test-pieces, simple plates were produced. For the bulge test casting trials, the waxes were connected together into a four-plate system with runners at the top (Figures 2 and 3) before preparing the ceramic moulds.



(a)

Figure 2: Wax plates for the bulge tests with fiducial lines and measurement positions.



(b)

Figure 3: Wax plates for bulge test assembled on runner supports.

3.3 Slurry preparation

Slurries were prepared by conventional mixing of the raw materials according to the recipes shown in Tables 4 and 5 using mixing tanks of the type shown in Figure 4.



Figure 4: Slurry mixing tank.

Some specific comments concerning the preparation of the slurries are as follows:

Full silica system: The primary slurry was very difficult to produce and use (thixotropic/thin layer); however, the secondary slurry was very stable and easy to control;

Full aluminosilicate: Secondary 1: slightly low initial viscosity, stable after additions; Secondary 2: very low initial viscosity, required large flour additions, stable;

Zircon/molochite and zircon/alumina: Secondary 1: very low initial viscosity, required large flour additions, thickened with time, gelled after completion of dipping;

Secondary 2 (Z/M): very low initial viscosity, required large flour additions, thickened with time.

The thicker mould systems 7, 8 and 9 were made using the same slurry formulations as the thinner ones, although 8 and 9 had slightly greater viscosities.

Table 4: Primary slurry system

Shell system	Mould type	Designation	Filler loading (kg/kg binder)*	Filler	Binder	Binder silica (%)	Polymer (%)	Antifoam (wt%)	Viscosity (secs B4)	pH
1	Full silica	NPL/2003/01	1.80	Min-Sil 120F	Remasol LP	27	6	0.3	100-120	9.4-9.8
2	Full aluminosilicate	NPL/2003/02	3.75	Zircosil 200 M	Remasol LP	27	6	0.3	100-120	9.4-9.8
3	Zircon / molochite	NPL/2003/02	3.75	Zircosil 200M	Remasol LP	27	6	0.3	100-120	9.4-9.8
4	Zircon / alumina	NPL/2003/02	3.75	Zircosil 200M	Remasol LP	27	6	0.3	100-120	9.4-9.8
5	Zircon / alumina	NPL/2003/02	3.75	Zircosil 200M	Remasol LP	27	6	0.3	100-120	9.4-9.8
7	Full silica 2	NPL/2003/01	1.80	Min-Sil 120F	Remasol LP	27	6	0.3	100-120	9.4-9.8
8	Full aluminosilicate 2	NPL/2003/02	3.75	Zircosil 200 M	Remasol LP	27	6	0.3	100-120	9.4-9.8
9	Zircon/ molochite 2	NPL/2003/02	3.75	Zircosil 200M	Remasol LP	27	6	0.3	100-120	9.4-9.8

* Density compensated for equivalent volume fraction loading

Table 5: Secondary slurry system

Shell system	Mould type	Designation	Filler loading (kg/kg binder)*	Filler	Binder	Binder silica (%)	Polymer, (%)	Viscosity (secs B4)
1	Full silica	NPL/2003/03	0.70	Min-Sil 120F	Remasol SP	22	8	30-35
2	Full aluminosilicate	NPL/2003/04	0.84	Molochite -200	Remasol SP	22	8	28-30
3	Zircon / molochite	NPL/2003/05	1.40	Zircosil 200M	Remasol SP	22	8	30-35
4	Zircon / alumina 1	NPL/2003/05	1.40	Zircosil 200M	Remasol SP	22	8	30-35
5	Zircon / alumina 2	NPL/2003/05	1.40	Zircosil 200M	Remasol SP	22	8	30-35
7	Full silica 2	NPL/2003/03	0.70	Min-Sil 120F	Remasol SP	22	8	30-35
8	Full aluminosilicate 2	NPL/2003/06	0.84	Molochite -200	Remasol SP	22	8	45-50
9	Zircon / molochite 2	NPL/2003/07	1.4	Zircosil 200M	Remasol SP	22	8	45-50

* Density compensated for equivalent volume fraction loading

3.4 Shell build, curing and dewaxing processes

The shells were built by first investing the wax pattern into the primary slurry. The stucco was applied by the rainfall sanding method. The coarse grains pass through a suspended agitated mesh and fall from a 1 m height as a shower onto the primary slurry coat. The assembly is rotated during this process in order to obtain a uniform coating of stucco material adhering to the slurry coating. The coating was dried at 21.5 °C, 50% RH and 0.4 m s⁻¹ air speed for 24 h. The back-up coats were then applied sequentially in the same manner. Each secondary coat was dried under similar conditions, but at 3 m s⁻¹ air speed for 90 minutes only. No final seal coat was applied. Figure 5 shows examples of the shell moulds produced.

The wax inside the mould was removed by steam autoclaving in a Quicklock Boilerclave (Leeds and Bradford Boiler Co., UK) at 8 bar maximum pressure for 4 min followed by a depressurisation cycle at 1 bar/min.

Table 6 summarises the codes used for each mould type, and Table 7 summarises the number of sets of moulds of each compositional type prepared in this programme. Figure 5 shows examples of dipped moulds for various test-piece types and the metal casting moulds.

For routine mould characterisation purposes at UoB, test-pieces cut from moulds were mostly fired at 1000 °C for 1 h.



Figure 5: Dipped moulds, from top left clockwise: MOR bars, plates, bulge test moulds, permeability moulds.

Table 6: Generic mould test-piece codes

Sample ID	NPL code	Date	Sample description	Generic mould ID	Firing profile*
NPL 1	PJW	23-Jan-04	Flat bar plates	NPL Shell System 1	none
NPL 2	PJX	23-Jan-04	MOR test pieces	NPL Shell System 1	none
NPL 3	-	23-Jan-04	40mm perm	NPL Shell System 1	standard
NPL 4	PJY	09-Feb-04	Flat bar plates	NPL Shell System 2	none
NPL 5	PJZ	09-Feb-04	MOR test pieces	NPL Shell System 2	none
NPL 6	-	10-Feb-04	40mm perm	NPL Shell System 2	standard
NPL 7	PKF	08-Apr-04	Flat bar plates	NPL Shell System 3	none
NPL 8	PKG	08-Apr-04	MOR test pieces	NPL Shell System 3	none
NPL 9	-	21-Apr-04	40mm perm	NPL Shell System 3	standard
NPL 10	PKH	22-Apr-04	Flat bar plates	NPL Shell System 4	none
NPL 11	PKJ	22-Apr-04	MOR test pieces	NPL Shell System 4	none
NPL 12	-	22-Apr-04	40mm perm	NPL Shell System 4	standard
NPL 13	PKL	07-May-04	Flat bar plates	NPL Shell System 5	none
NPL 14	PLM	07-May-04	MOR test pieces	NPL Shell System 5	none
NPL 15	-	10-May-04	40mm perm	NPL Shell System 5	standard
NPL 16	PKS	27-Jul-04	Flat bar plates	NPL Shell System 8	none
NPL 17	PKT	27-Jul-04	MOR test pieces	NPL Shell System 8	none
NPL 18	-		40mm perm	NPL Shell System 8	standard
NPL 19	PKX	23-Aug-04	Flat bar plates	NPL Shell System 9	none
NPL 20	PKY	23-Aug-04	MOR test pieces	NPL Shell System 9	none
NPL 21	-	25-Aug-04	40mm perm	NPL Shell System 9	standard
NPL 22	PKZ	02-Sep-04	Flat bar plates	NPL Shell System 7	none
NPL 23	PLA	02-Sep-04	MOR test pieces	NPL Shell System 7	none
NPL 24	-	02-Sep-04	40mm perm	NPL Shell System 7	standard

* Standard furnacing profile for casting moulds: 0 - 700 @ 2°C/min, dwell = 6 mins, 700 °C-1000 °C @ 20°C/min, dwell = 60 mins , furnace cool

Table 7: Moulds made at University of Birmingham for each compositional type

Test-piece type	Form	Number dipped
Strips for MOR bars for NPL	Double sided	10
Strips for MOR bars for UoB	Double sided	10
Flat plates for NPL	Double side	1
Permeability moulds for UoB	Spherical	3
Edge integrity test-pieces for UoB	Right-angles	3
Full scale bulge assemblies for RR	Four plates in each	2

4. TESTING METHODS

4.1 University of Birmingham (UoB)

4.1.1 Flexural strength (MOR)

Flexural strength tests were made in three-point bending over a span of 50 mm using test-pieces of dimensions approximately 80 mm x 20 mm x mould thickness cut from the MOR bar mouldings by diamond wheel sawing. Testing was conducted in an Instron 8500 universal testing machine with a 5 kN load cell. A cross-head displacement rate of 1 mm/min was used. Tests were conducted on:

1. “dried” test-pieces (24 h at room temperature after autoclaving);
2. “wet” test-pieces (placed over a steam bath for 15 min before testing);
3. “fired” test-pieces (1000 °C for 1 h).

Results were calculated as nominal flexural strength in MPa using the usual linear elastic formulation:

$$\sigma_f = \frac{3F_{\max}L}{2WH^2} \quad (1)$$

where $F_{\max}$ is the fracture force, L is the test span, W is the test-piece width, H = test-piece height. The load deflection curves were converted to stress strain curves using the relationships:

$$\sigma = \frac{3FL}{2WH^2}, \quad \varepsilon = \frac{6H\delta}{L^2} \quad (2)$$

where δ is the central deflection. Young’s modulus was computed from the stress/strain curves by linear regression. No correction was made for load-train stiffness.

The ‘adjusted fracture load’ in flexure (AFL_B) is defined as the force required to break a 10 mm wide test-piece of shell across a 70 mm span, and is a method of normalising the load bearing capacity of the shell. It is expressed as:

$$AFL_B = f_B \sigma_{\max} H^2 \quad (3)$$

where f_B is a constant equal to 0.10.

4.1.2 Edge strength

The edge strength test has been developed by UoB as a quality control procedure to determine the actual strength of the shell at the most vulnerable edges where strength and shell build are vital to prevent cracking during de-waxing and casting. Instead of a plane mould surface, a 29° wedge is forced into a Vee-shaped test-piece (see Figure 6). The test-piece is loaded such that the inner surface of the mould (i.e. the primary layer) is in tension across the edge, and the outer surface is in compression. The force required to break the test-piece is recorded, and the edge strength is calculated using:

$$\sigma_{edge} = 12.2 \frac{Fd \sin \theta \cos \theta}{WT^2} \quad (4)$$

where d is the span length, θ is the included wedge angle, W is the test-piece width and T is the test-piece edge thickness. The adjusted fracture force (AFL_w) is defined as the force necessary to break a 10 mm wide test-piece with a 20 mm span length, which normalises the force-bearing capacity of shells at edges, and can be expressed as:

$$AFL_w = f_w \sigma_{edge} T^2 \quad (5)$$

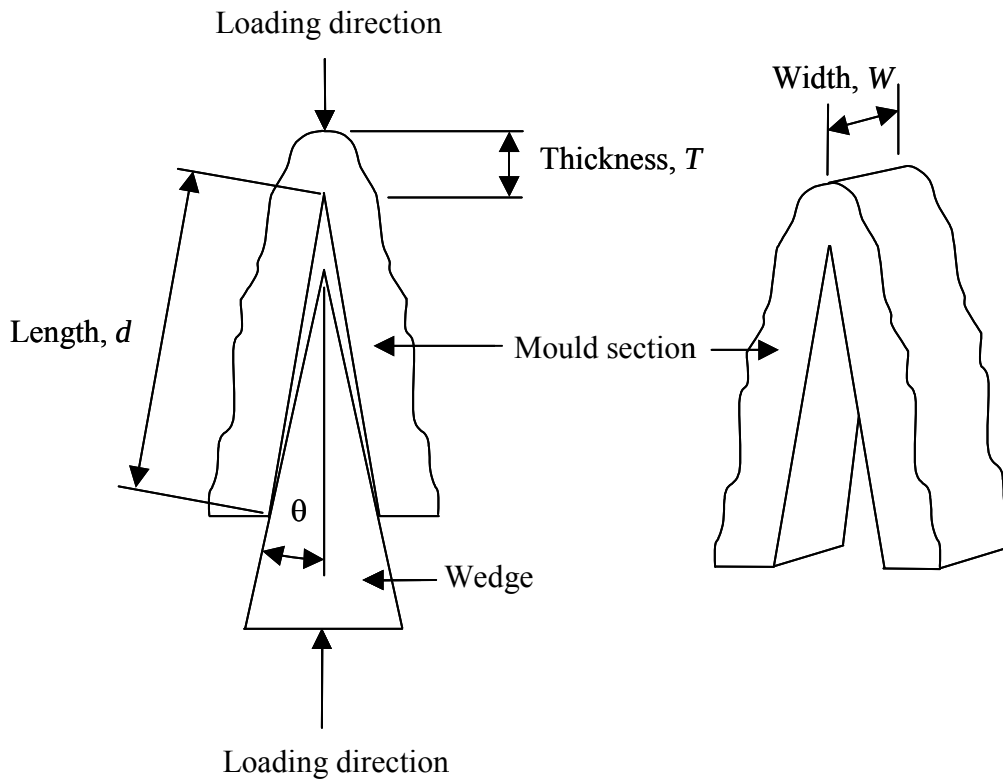


Figure 6: Schematic arrangement for the edge strength test.

4.1.3 Permeability

Permeability tests were conducted in accordance with BS1902 Part 10.2 using the moulded test-pieces shown at the lower left in Figure 5. The volumetric flow rate of air is measured for a given pressure drop through a known wall thickness. Results are calculated in units of m^2 using the expression:

$$\mu = \frac{\eta V_c l}{aP} \quad (6)$$

where η is the temperature dependent viscosity from a polynomial fit with known coefficients, V_c is the volumetric flow rate of air in m^3/s , l is the thickness of the mould in metres, a is the surface area of the mould in m^2 , and P is the pressure difference across the test-piece in Pa.

4.1.4 Plate bulge test

The principle of the plate bulge test was taken from an earlier proposal from work undertaken by the UK National Engineering Laboratory (5). Flat plate wax formers were scribed with a rectangular pattern of fiducial lines (Figures 2 and 7(a)) which could be used to define a grid of measurement positions. The lines were then incorporated as ridges on the metal contact face of the mould, and after casting were copied as a pattern of grooves in the cast metal plate. Wax former and metal plate thicknesses at each grid intersection were measured using a micrometer. The difference in thickness at each point was computed and used to establish whether the mould surface had bulged (assumed symmetrically). The measurements could not be made down the middle zone of the plates owing to the lack of availability of a suitably wide span micrometer.

4.2 National Physical Laboratory

4.2.1 Dimensional changes on sintering

Rectangular test-pieces of side between 25 and 50 mm of each green (unfired) mould material were cut using a diamond saw, and the edges were ground square, flat and parallel within 0.2 mm. Three measurements were made (Figure 8(a)) using calibrated digital callipers in both lateral directions (parallel and perpendicular to the vertical direction during mould manufacture). The test-pieces were then placed on a flat insulating firebrick in a Lenton 1700 °C capability furnace with a temperature programmer, and fired with the following schedule: RT – 1000 °C @ 10 °C/min; hold 1000 °C / 2 h; 1000 °C to 1250 °C, 1350 °C or 1450 °C @ 10 °C/min with a 1 h hold, followed by furnace cooling. An additional set of tests was made with a hold at 1000 °C / 10 h to simulate the scenario where a mould might be left in the pre-heat furnace for a protracted period. When cold, the dimensions were re-measured at the same positions, and the average size change determined.

It was noted that during the flexural creep tests (see below) some materials tended to curve out of plane during heating. It was presumed that this would be a result of differential shrinkage between the various layers of the structure. To investigate this aspect, strip test-pieces typically 120 mm to 140 mm in length were cut. Any undulations in the metal contact face were carefully removed by minimal grinding on a flat lap. The test-piece was then characterised for curvature along the mid-line of the metal contact face by placing it stucco side down on the bed of a height-measuring microscope. The height profile of the curved test-piece was determined by focusing on the surface at a nominal 100x magnification to a height resolution of 0.001 mm at seven locations as shown in Figure 8(b). The test-pieces were then fired in the same way as the rectangular section test-pieces above, but they were set on the firebrick resting on an edge to minimise the gravitational effects. When cold, they were subjected to the same height measurement procedure, locating the same positions as closely as possible, allowing for any longitudinal shrinkage.

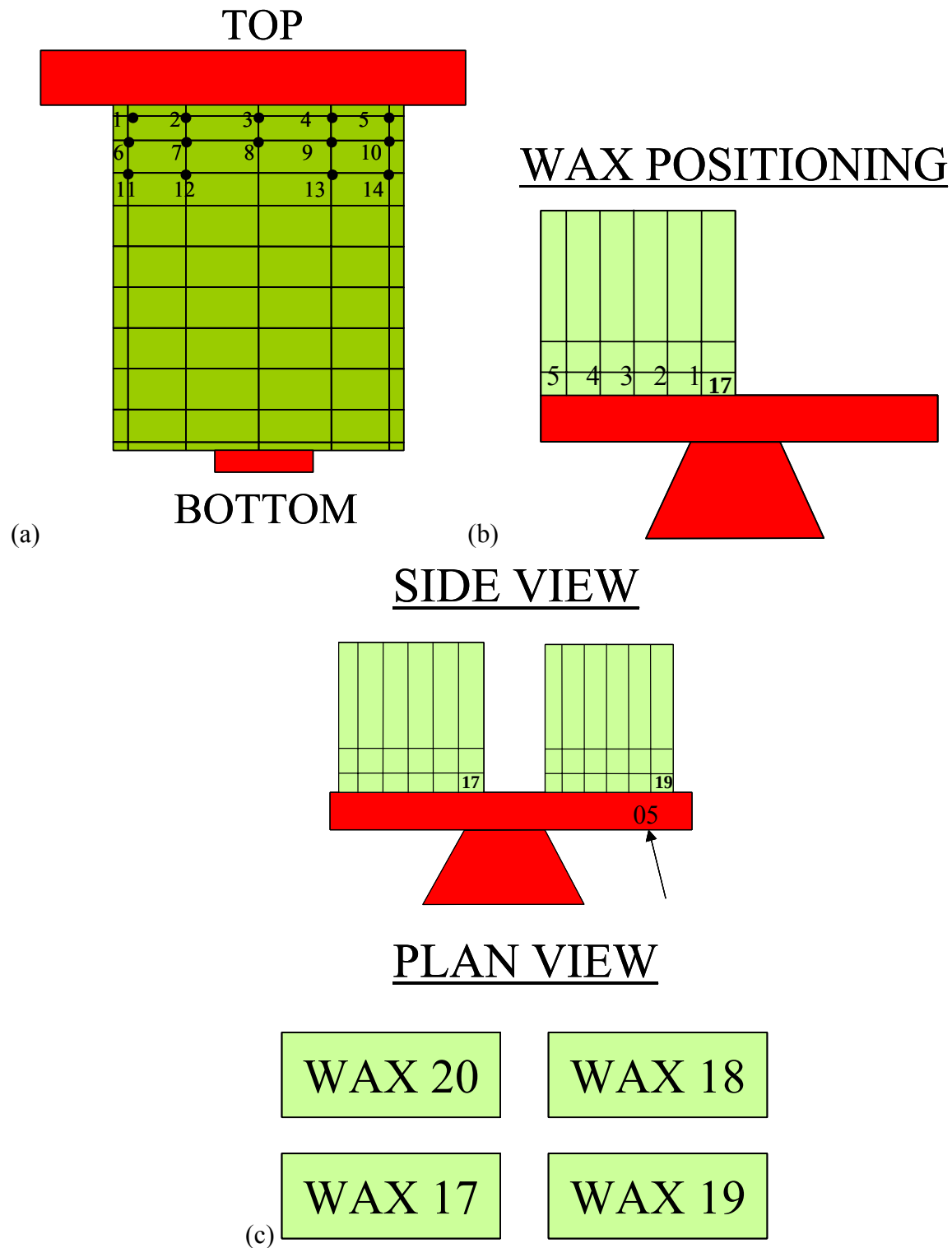


Figure 7: Manufacture of waxes for the plate bulge test. (a) fiducial lines and measurement positions on individual plate, (b) wax plate no. 17 fixed to runner system shown inverted, (c) four plates fixed to runner system, each plate being identified by a plate number (in this case nos. 17-20), and the mould type (in this case 05) being inscribed on the runner. Two runner sizes were used, each with two waxes of nominal thicknesses 7.5 mm and 13 mm.

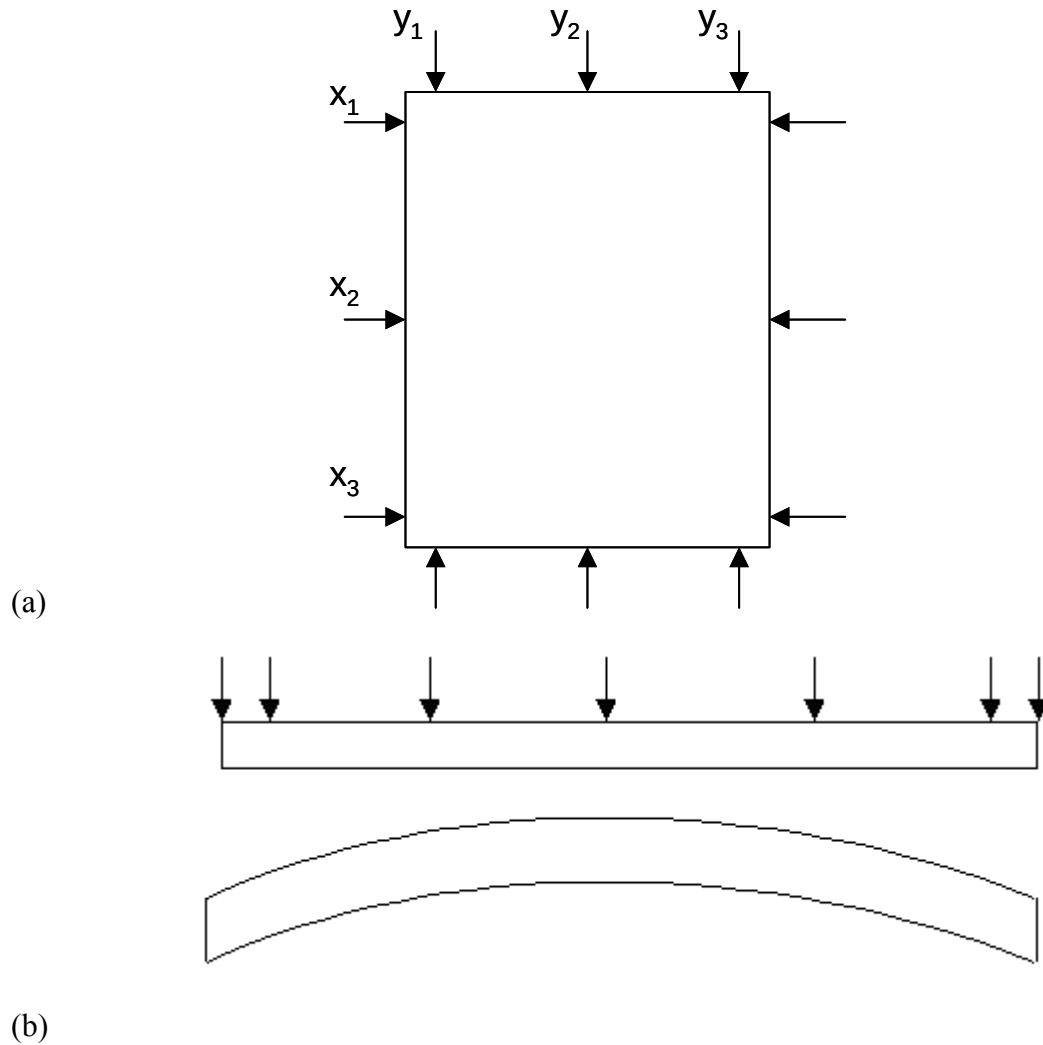


Figure 8: Dimensional measurement methods (a) on a rectangular tile with y parallel to the vertical direction in manufacture and x perpendicular to this direction; and (b) strip curvature with height position measurements indicated, all measurements with metal contact face uppermost.

The data from the strip curvature measurements were processed as follows. For both unfired and fired conditions, the raw height data were first plotted. The height difference between the two ends was used to provide a levelling correction to the data (necessary because of the rough, as-manufactured stucco side being in contact with the microscope base-plate). A parabolic fit was then made to these corrected data to approximate to a circle of large radius, and the radius of curvature was determined as:

$$\rho = 1 / \left(\frac{d^2 y}{dx^2} \right) = 1 / 2a \quad \text{where} \quad y = ax^2 + bx + c \quad \text{is the fitted height equation.} \quad (7)$$

The data for the fired strip were processed in a similar manner, and the differences in curvature, $1/\rho$, determined as a measure of the net curvature produced by the firing process.

4.2.2 Thermal expansion measurement

The measurements were made in a Linseis dilatometer with specially fabricated alumina apparatus. The thermal movement of the test-piece relative to the work-tube is recorded by the instrument's displacement transducer. The work-tube and test-piece were surrounded by a SiC element tube furnace, with constant ramp rate temperature control. The temperature of the test-piece was recorded separately from the tube furnace temperature, using a type R thermocouple in contact with the test-piece surface. The instrument had been previously calibrated in the following manner. The absolute sensitivity of the displacement transducer was determined using a drum micrometer which is placed in line with the push-rod after removal of the work-tube. The baseline shift response of the apparatus was determined by moving the push-rod to contact the apparatus end-stop, and performing thermal cycles. The correction for the expansion of the apparatus was determined by making thermal cycles with a NIST certified silica reference material SRM 739 and with a platinum reference bar. The data from the baseline and reference material thermal cycles were processed using an EXCEL spreadsheet to provide polynomial curvefits for the respective corrections to be employed on a test-piece with unknown characteristics. This procedure is documented in NPL/DMM/B119, and follows the principles laid down in BS EN 821-1 and ASTM E228. The uncertainty in the calibration as applied to a mechanically stable and repeatably behaving test-piece is considered to be approximately $\pm 2\%$ in fractional length change or $\pm 0.2 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ in expansion coefficient over a 100 $^{\circ}\text{C}$ range, whichever is the greater.

The initial test-piece length was measured to the nearest 0.01 mm using a calibrated micrometer.

Transducer displacement and test-piece thermocouple voltages are logged at regular intervals using a data logger. The resulting data set from measuring an unknown was processed using an off-line program which converts it to absolute fractional change in length in parts per million from a reference temperature of 25 $^{\circ}\text{C}$ using the polynomial calibration functions, and the set then plotted. Fractional changes in length at particular temperatures were computed by linear interpolation between logged datapoints.

For the purposes of the present tests, a heating/cooling rate of 2 $^{\circ}\text{C}/\text{min}$ was mostly used, with data being logged every two minutes. A single thermal cycle only was performed on each test-piece. Faster heating or cooling results in significant apparatus hysteresis, and is generally avoided

4.2.3 Flexural creep tests

These tests were carried out in a Severn Science flexural creep apparatus based on a split box furnace surrounding an alumina tube pillar on which the flexural loading jig was placed. This was made from a flat alumina plate with two 10 mm diameter alumina rollers spaced by two alumina strips. This arrangement ensured the use of a consistent 77 mm span⁴ and freely rolling rollers to accommodate flexure without friction. The load was applied through a centrally placed alumina loading roller on which a flat had been machine. Because the forces required were so low, a 12 mm diameter closed-end alumina tube was used as the loading pillar, the rounded end of which contacted the flat on the loading roller (Figure 9). This arrangement permitted a self-alignment capacity. A weight carrier was constructed to fit onto the cold end of the loading tube.

⁴ This span is within the tolerance band of $76 \pm 1 \text{ mm}$ for the deformation test described in BS 1902 part 10.1.

The flexural displacement was measured using a pair of water-cooled displacement transducers mounted below the alumina support tube and operated in differential mode with 1.5 mm diameter alumina extensometer rods. One rod passed through a slot in the alumina plate to contact the centre of the flexing test-piece. The other contacted the underside of the plate. This arrangement has the advantage that most of the effects of thermal expansion of the extensometer rods during heating are removed. Figure 9 shows the general arrangement of the facility. The transducer system was calibrated over the differential voltage range used with a depth micrometer moving one extensometer tip while the second was contacting the flat micrometer reference surface. Data comprising apparent flexural displacement and temperature from a type R thermocouple with its tip close to but not touching the test-piece were logged using a computer data logger at typically 60 s or 1 s intervals depending on the type of test.

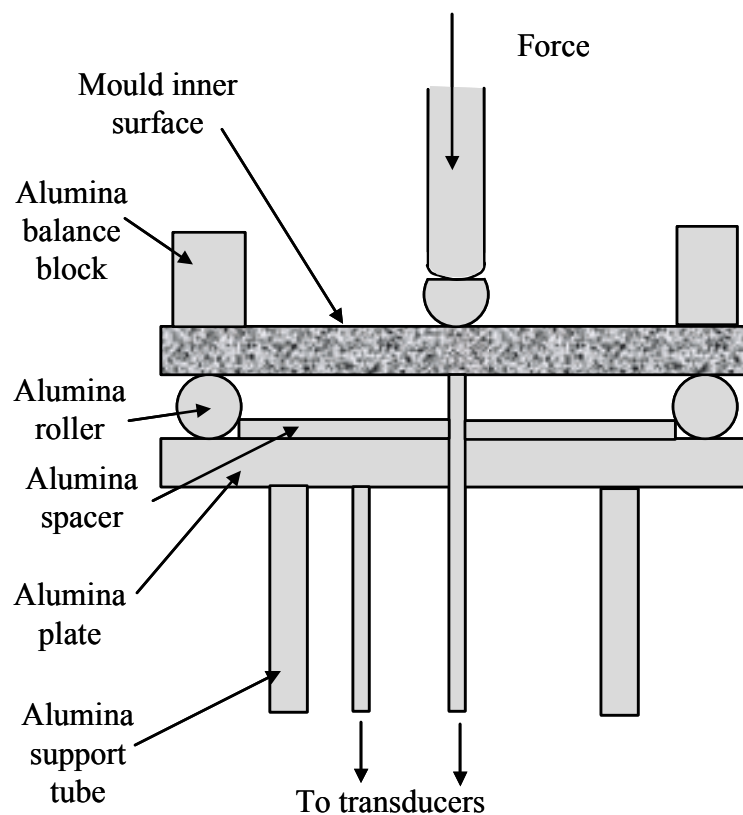


Figure 9: Arrangement for flexural creep testing.

Test-pieces were prepared as flat strips cut from the supplied mould materials to be typically 100 mm in length, 10-15 mm in width and 4-9 mm in thickness. The sides were hand-ground to be flat and parallel to within 0.1 mm. The metal contact face was gently flattened on a flat SiC paper lap. Using a diamond impregnated flat lap, the stucco side was flattened by an amount necessary to create flat and parallel faces for loading the test-piece. Because some of the supplied materials were not of even thickness, this necessitated the removal in some cases of up to 1 mm of material from parts of the surface. Test-piece lateral dimensions were measured to the nearest 0.01 mm using a micrometer.

The test force was calculated for a given outer-layer tensile stress level at the centre of the test-piece opposite the loading roller using the three-point flexure equation for elastic conditions. The mass required to be added to the dead mass of the loading tube was then calculated, and this mass was prepared using lead shot in a plastic box to be placed on the carrier on the end of the loading tube.

The test procedure was as follows:

1. Place the test-piece across the support rollers and ensure its stability. Position the support rollers against the spacer.
2. Place the loading roller flat side up across the centre line of the test-piece.
3. Position two small alumina balance blocks on the ends of the test-piece above the support rollers to counteract the extensometer force.
4. Raise the extensometers to contact the plate and test-piece and adjust the position to ensure an appropriate starting position on the output voltage scale.
5. Position the loading tube at least 3 mm above the central loading roller to allow for thermal expansion during heating, and add the required mass.
6. Close the furnace and apply the temperature programme required.
7. After the final temperature was achieved, allow to soak for 2 min, and then release the loading tube to contact the loading roller.
8. Record the creep curve at a 1 s logging interval for 4 min.
9. Cool down.

A standard temperature programme was used as follows:

Heat at 20 °C/min to 1000 °C and hold 2 h

Heat at 10 °C/min to the test temperature of 1250 to 1450 °C and hold for the test duration.

In some cases the test force was applied from cold.

Data were processed using a spreadsheet permitting plots of apparent flexural displacement or strain with time to be plotted. The central displacement δ is related to the flexural strain ε as:

$$\varepsilon = \frac{6H\delta}{L^2} \quad (8)$$

where H is the test-piece thickness and L is the support roller span, assuming viscous behaviour.

4.2.4 Compression creep

Compression creep tests were undertaken in the same apparatus as described above. Because the force required to achieve a given compression stress is much higher than for the flexural geometry, the linear bearing guided alumina ram of the original flexural creep apparatus was employed, together with the lever loading system.

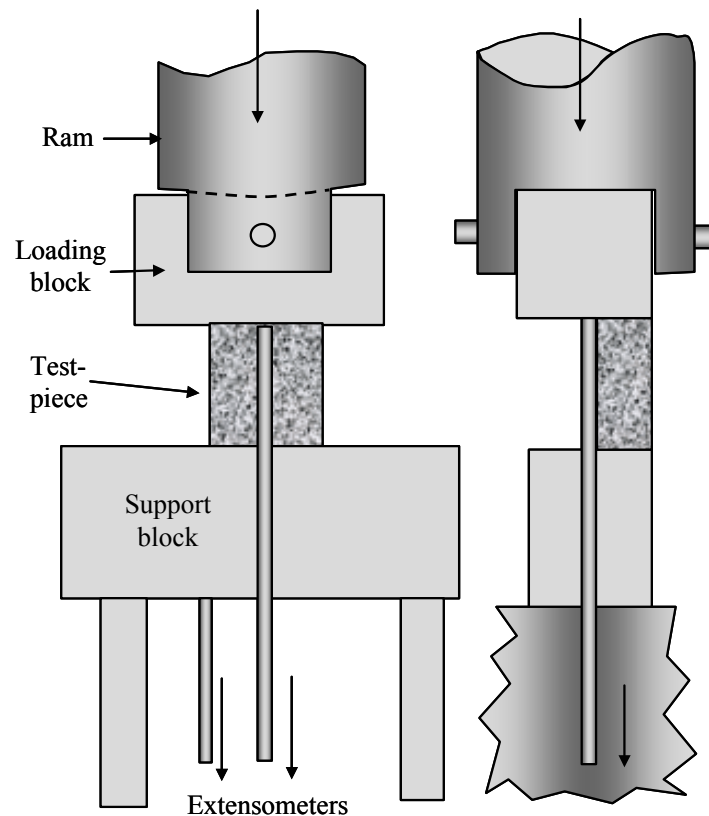


Figure 10: Arrangement for compression creep testing.

The test-piece was stood vertically on an alumina block and loaded by a similar block flexibly pinned into the end the loading ram. The extensometer system passed through a hole in the support block to contact the loading block adjacent to the test-piece. The arrangement is shown schematically in Figure 10. Although this arrangement was not coaxial, it functioned well because of the guided ram and rigid support system.

Test-pieces approximately 15 mm in length by 12 mm wide and four to six mm thick were prepared by cutting and grinding to give ends flat and parallel to within 0.05 mm. The test-piece thickness was limited by the nature of the loading block, and to achieve this, material was removed from the stucco side of the thicker materials.

The test force was computed based on the cross-sectional dimensions of the test-piece, and a suitable mass was applied to the loading pan suspended from the end of the 5:1 lever loading arm after correcting for the mass of the ram system (≈ 4.41 N) which was applied at all times to the test-piece.

4.2.5 Flexural strength

A few high-temperature flexural strength tests were performed in a similar furnace system sited within the frame of an Instron universal mechanical testing machine. The same flexural strength jig was employed, but without the extensometers, the deflection being based on machine displacement at constant cross-head displacement rate. The loading ram was of sintered silicon carbide and, to ensure alignment, a cylindrical Vee-notched jockey was employed between the loading roller and the flat end of the ram. A cross-head displacement rate of 1 mm/min was employed.

5. MATERIALS TESTING RESULT

5.1 Cutting of test-pieces and mould cross-sections

While it was straightforward to prepare green-state test-pieces by diamond saw-cutting and flat lapping, the materials being easy to machine, there were limitations to the quality of cut in some, particularly those such as Generic 2 and 8 (both aluminosilicate) in which the larger stucco particles were only weakly bonded. These particles were readily plucked out, leaving pits and causing some additional damage. This damage could not be reliably removed by fine lapping because pluck-out would continue. Otherwise, there were generally no problems in preparing the long narrow flexural test-pieces to the required dimensional precision.

Cross-sections of moulds were prepared for microscopy by subjecting them to a final 'polish' using 600 grit SiC paper on a flat lap. These cross-sections are shown in Figure 11. In each case the metal contact face is at the lower side of the image. The stucco side is generally very rough, and only in one or two cases is a layer structure obvious.

5.2 Granulometry

This activity was organised by Minco UK Ltd. Images of the coarser stucco materials are given in Annex 2. Sieve fraction analysis was conducted on all materials (after removal of fines in the four finest materials) and particle size analysis was conducted on the finer grained materials using a MicroTrac instrument. Results are given in Annex 2.

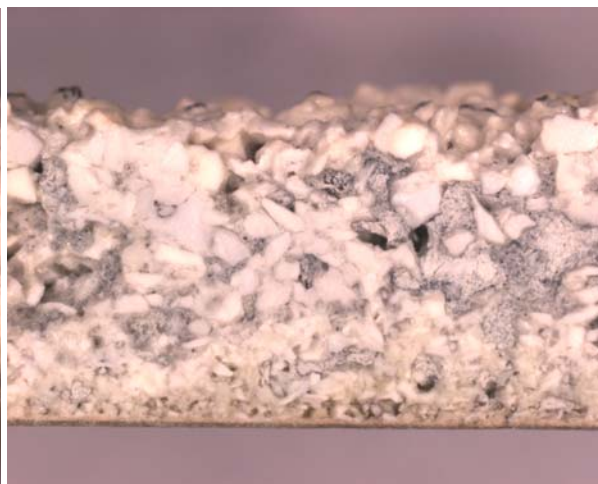
5.3 Flexural strength

Flexural strength tests were performed on test material in three conditions: unfired dry, unfired wet, and fired to 1000 °C. The bars varied in thickness as shown by Figure 12 (a), but this is effectively normalised in calculation of strength (Figure 12(b)). It can be seen that the full silica system (Generics 1 and 7) is the weakest in the wet and fired conditions, probably as a result of its low density and high permeability. The full aluminosilicate is a little stronger, but the zircon/molochite and zircon/alumina systems (Generics 3 and 9, Generics 4 and 5 respectively) are significantly stronger under all conditions.

Figure 13(a) shows the adjusted fracture load, which is the fracture force that would be obtained for a given 10 mm width over a 70 mm span. Figure 13 (b) shows the effective elastic moduli computed from the slope of the load displacement curves in three-point bending. It is clear that the zircon/molochite and zircon alumina systems are the stiffest by a substantial margin, but interestingly are not stiffened further by firing to 1000 °C. The least stiff is the full silica system, with the full aluminosilicate intermediate.



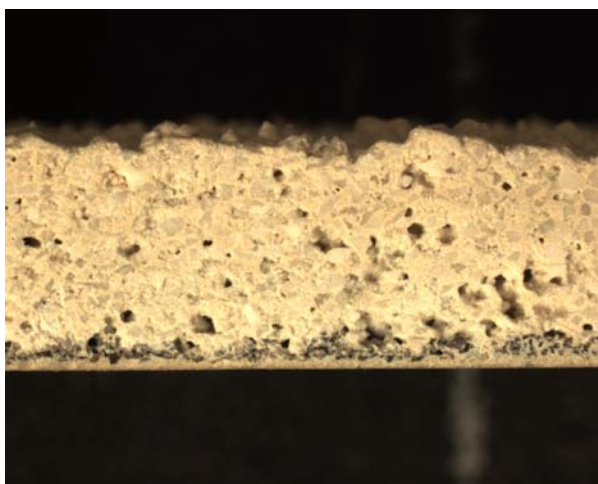
Generic 1, full silica 1, code PJX



Generic 2, full aluminosilicate 1, code PJZ



Generic 3, zircon/molochite, code PKG



Generic 4, zircon/alumina 1, code PKJ



Generic 5, zircon/alumina 2, code PKM



Generic 7, full silica 2, code PLA

Figure 11: Mould cross-sections. In all the above pictures, the height of the field of view is approximately 12 mm. Note that Generic 7 is a thicker version of Generic 1.



Generic 8, full aluminosilicate 2, code PKT



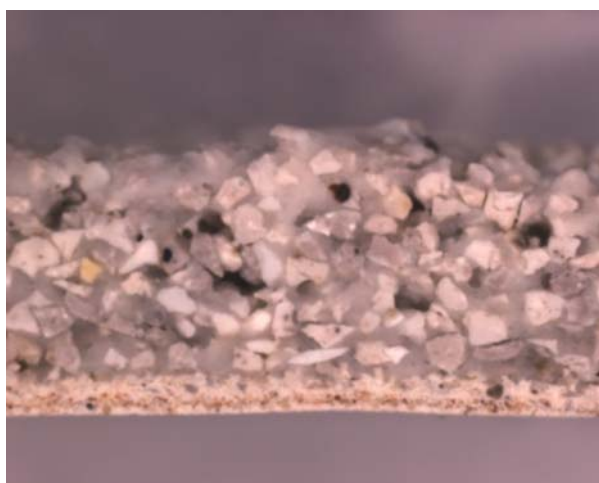
Generic 9, zircon/molochite 2, code PKY



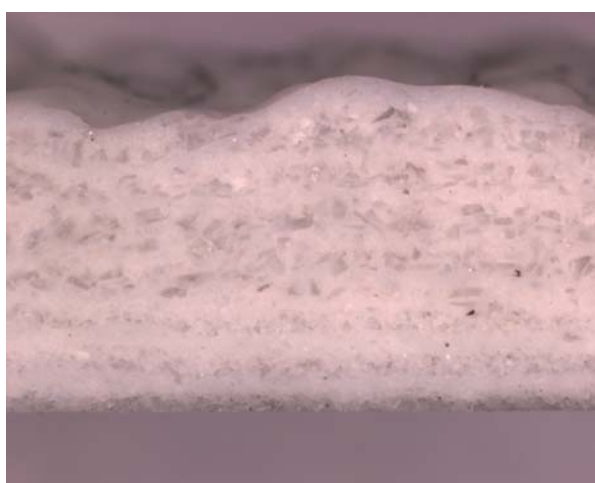
Partner 1, code PJS



Partner 2, code PJU

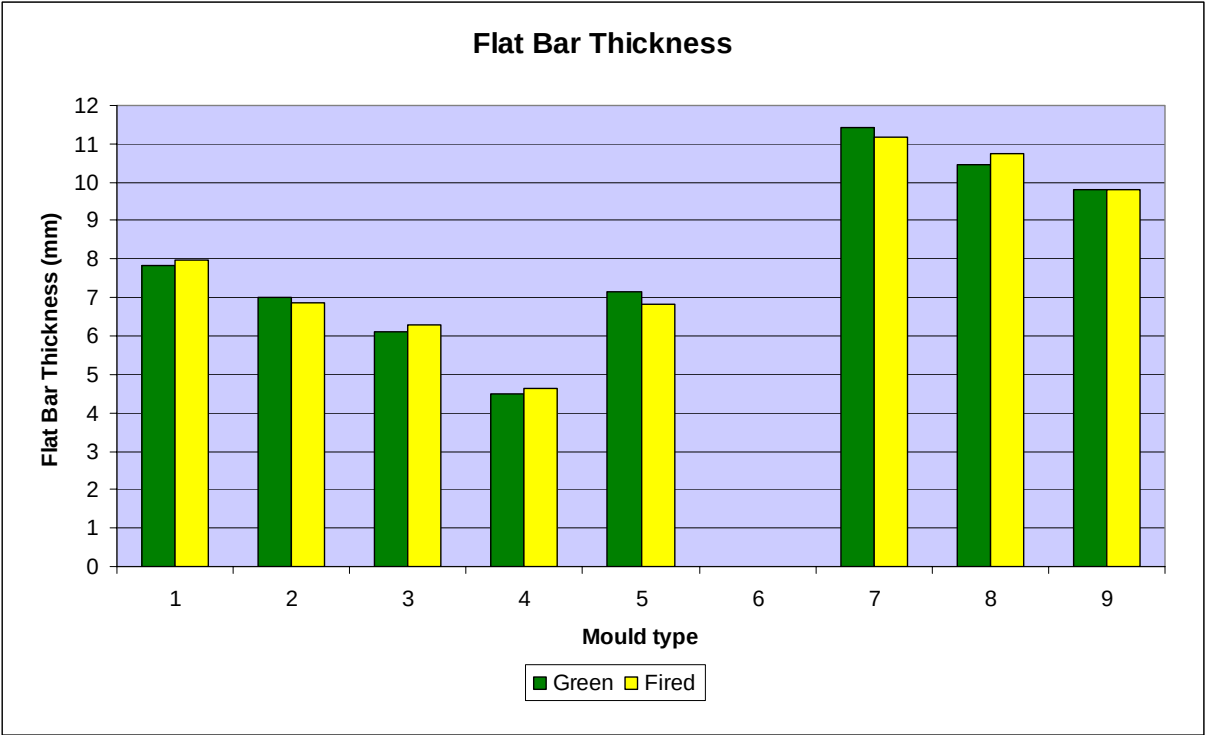


Partner 3, code PJV

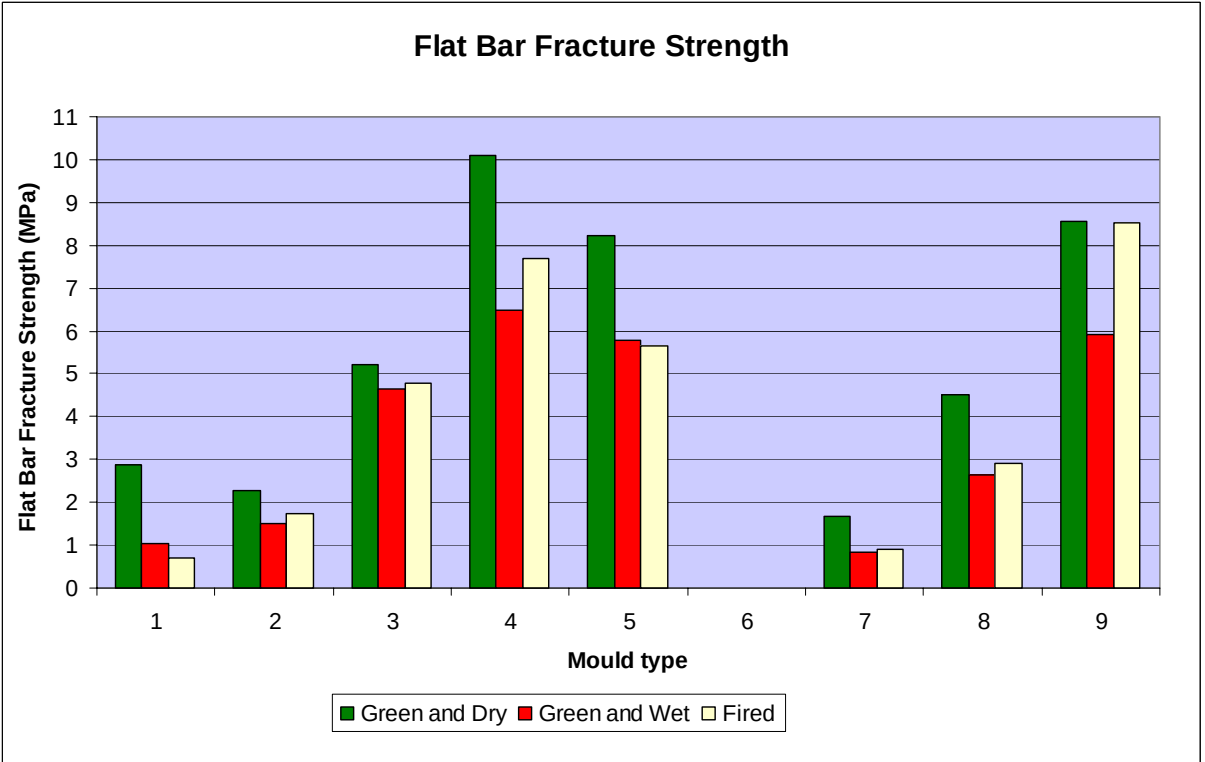


Partner 4, code PKA

Figure 11 (cont.): Mould cross-sections. In all the above pictures, the height of the field of view is approximately 12 mm. Note that Generics 8 and 9 are thicker versions of Generic 2 and 3 respectively.

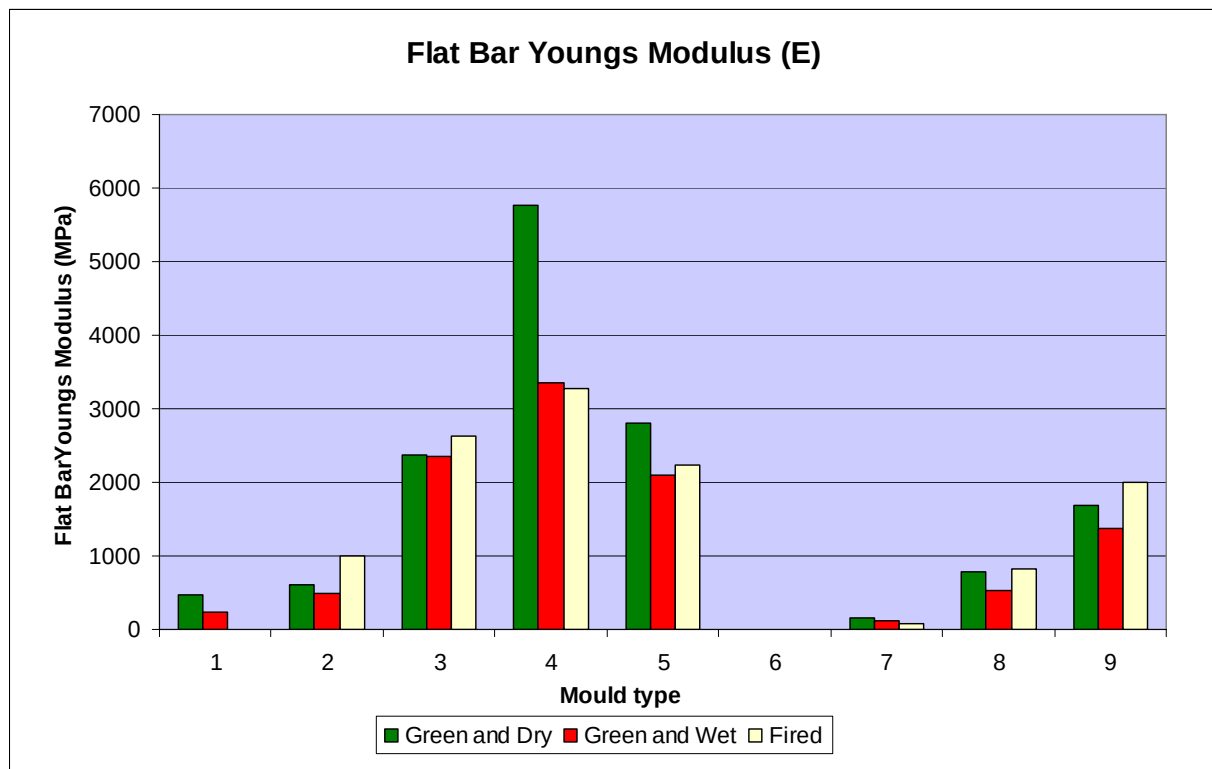


(a)

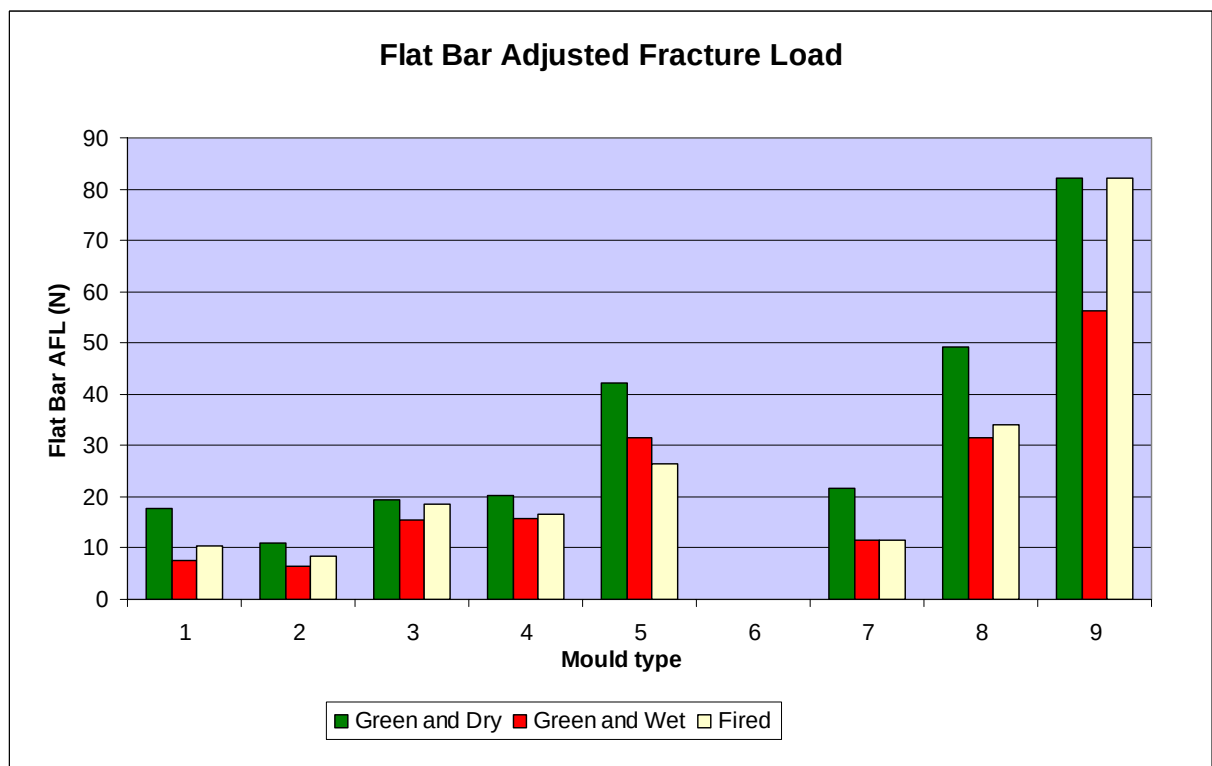


(b)

Figure 12: (a) Flat bar thickness for the different generic moulds used for flexural strength measurements, and (b) mean flexural strengths recorded by University of Birmingham.



(a)



(b)

Figure 13: (a) adjusted fracture load for each of the generic moulds, and (b) the Young's modulus estimated from the slope of the elastic regions of the load/displacement curves.

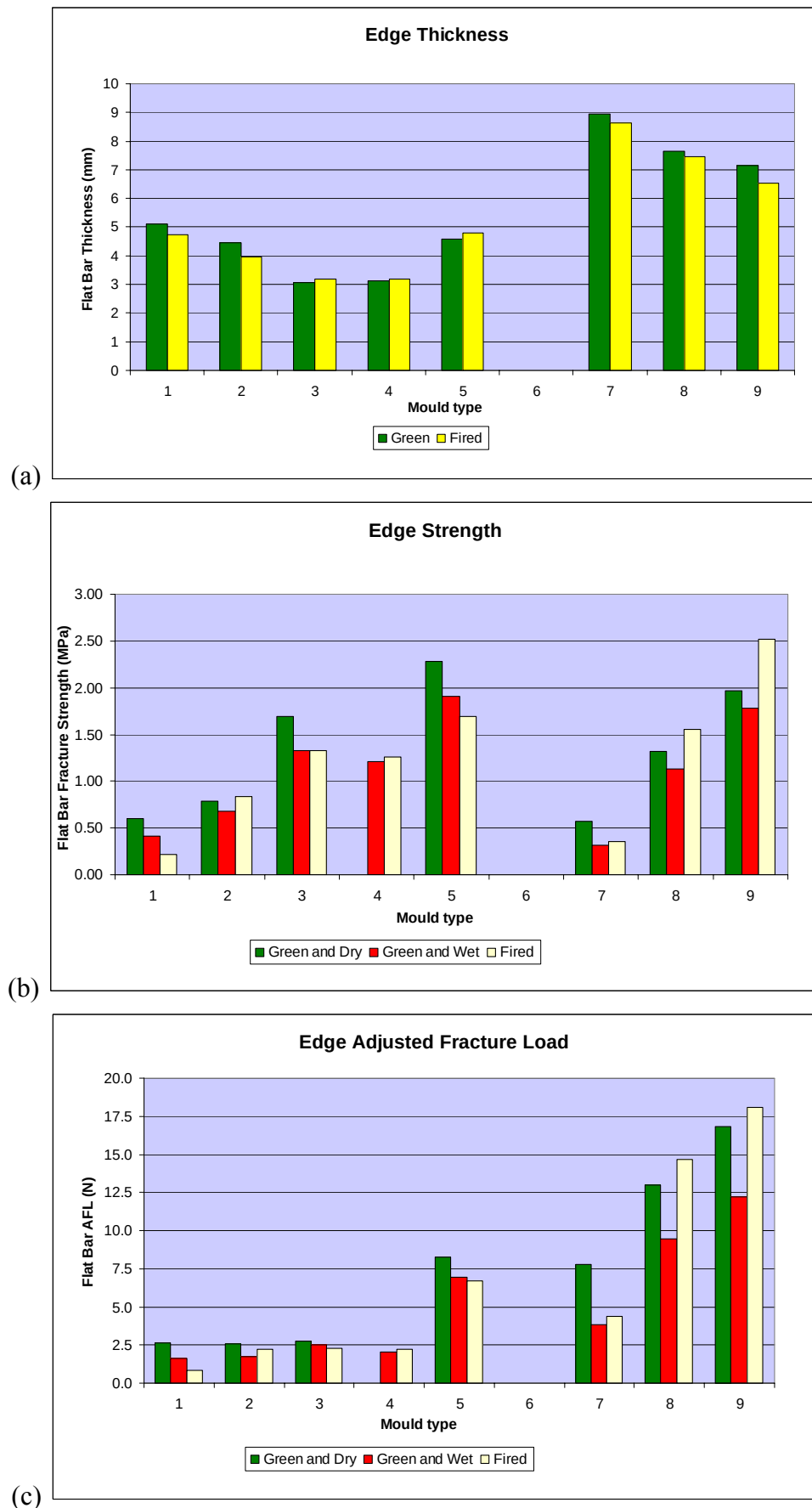


Figure 14: (a) Edge thickness, (b) edge strength, and (c) adjusted fracture load for the edge strength test. Note the significant improvements in moving to the thicker Generics 7, 8 and 9.

5.4 Edge strength

Figure 14(a) shows the thickness of the edges developed in the mould manufacturing process, and Figure 14(b) shows the strength of the edges obtained by the wedge loading method. The strongest moulds in terms of fracture stress in all three conditions, green and dry, green and wet, and fired to 1000 °C, are those based on alumina, while the weakest is the full silica system. Relevant to actual casting, the actual force required to fracture a 10 mm edge length is shown in Figure 14(c). It can be seen that the thicker moulds, Generics 7, 8 and 9, and also Generic 5, the mould type with the highest fracture stress in the flexural strength tests, all have strengths above 4 N, whereas the thinner moulds, Generics 1 to 4 have values rather lower than 4 N. It is considered that this test provides an indication as to the suitability of a given mould type and wall thickness for survival in the casting operation.

5.5 Permeability

This was tested in the fired condition and at room temperature only, using the method described in BS 1902 Part 10.2. The results are shown in Figure 15(a), which includes additionally a commercial mould (no.10). The results for Generics 1 and 7 were an order of magnitude higher than the other mould types. Such high values correlate with the low strength and modulus, and are indicative of a very low density.

Generic 4 has the lowest permeability, and since this was the strongest and stiffest mould (Figures 12(b) and 13(b) respectively), it was decided to investigate any correlation between permeability and strength. This is shown in Figure 15(b).

5.3 Dimensional changes on sintering

The results are given in Table 8. It can be seen that shrinkage generally always occurs, although it is minimal after firing to only 1000 °C for simulating the furnace pre-heat stage. It attains a little over 1% after firing to 1350 °C, and up to nearly 2% after a 1450 °C firing. It should be noted that the prolonged holds to which these samples were subjected is rather in excess of the duration normally experienced in casting, but it permits a ranking by material type to be made. This is more readily seen in Figure 16. The aluminosilicate moulds Generic 2 and 8, and the full silica moulds Generic 1 and 7 have by far the greatest shrinkage, and a similar outcome is shown by Partner 4.

There is no clear indication of a significant difference between shrinkages in the x and y directions when allowance is made for potential measurement inaccuracies of about 0.02 mm, equivalent to 0.05%.

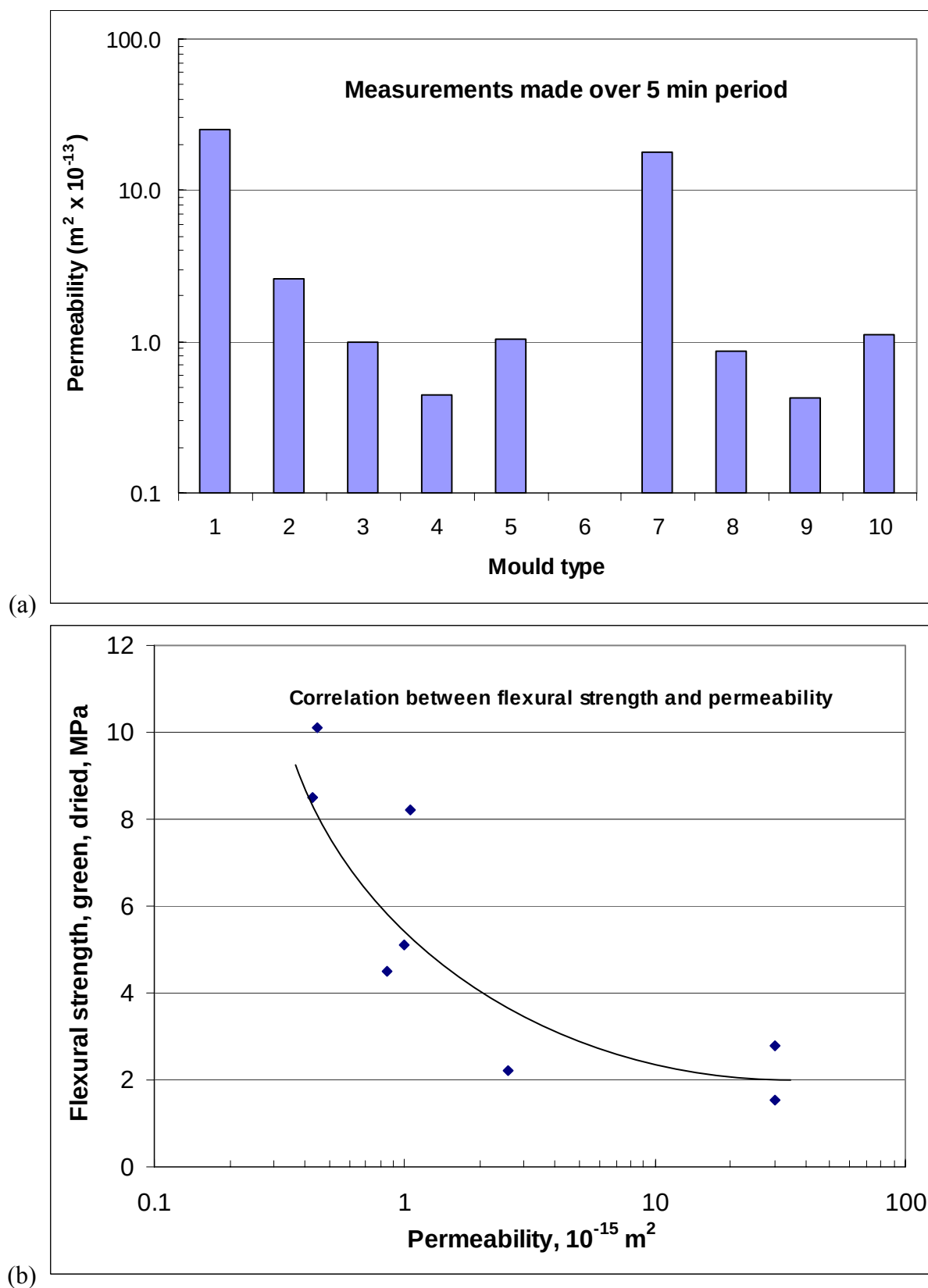


Figure 15: (a) Permeability at room temperature for the generic mould series; no. 10 is a commercial mould; and (b) a rough correlation between permeability and mould green, dried flexural strength.

Table 8: Lateral dimensional changes, fired flat

Directions: x = perpendicular to vertical direction of mould manufacture,
 y = parallel to vertical direction of mould manufacture

Mould code	Mould name	1000 °C/10 h	1000 °C/10 h	1350 °C/1 h	1350 °C/1 h	1450 °C/1 h	1450 °C/1 h
		Size change x , %	Size change y , %	Size change x , %	Size change y , %	Size change x , %	Size change y , %
PJS	Partner 1	0.05	0.00	-0.55	-0.35	-0.76	-0.34
PJU	Partner 2	-0.17	-0.26	0.00	-0.19	-0.92	-0.65
PJV	Partner 3	0.03	-0.11	-0.34	-0.17	-0.65	-0.12
PKA	Partner 4	-0.20	-0.13	-1.20	-1.24	-1.27	-1.08
PJW	Generic 1 (NPL1)	-0.04	-0.02	-1.08	-1.11	-1.32	-1.29
PJY	Generic 2 (NPL4)	-0.09	-0.20	-1.11	-0.99	-1.62	-1.49
PKG	Generic 3 (NPL7)	-0.19	-0.14	-0.43	-0.46	-0.87	-0.83
PKH	Generic 4 (NPL10)	-0.11	-0.12	-0.30	-0.19	-0.46	-0.39
PKL	Generic 5 (NPL13)	-0.02	0.01	-0.16	-0.13	-0.12	-0.28
PKZ	Generic 7 (NPL22)	-0.45	-0.33	-1.03	-0.95	-1.30	-1.25
PKS	Generic 8 (NPL16)	-0.16	-0.04	-1.01	-1.03	-1.88	-1.79
PKX	Generic 9 (NPL19)	-0.09	-0.16	-0.46	-0.42	-1.07	-1.12

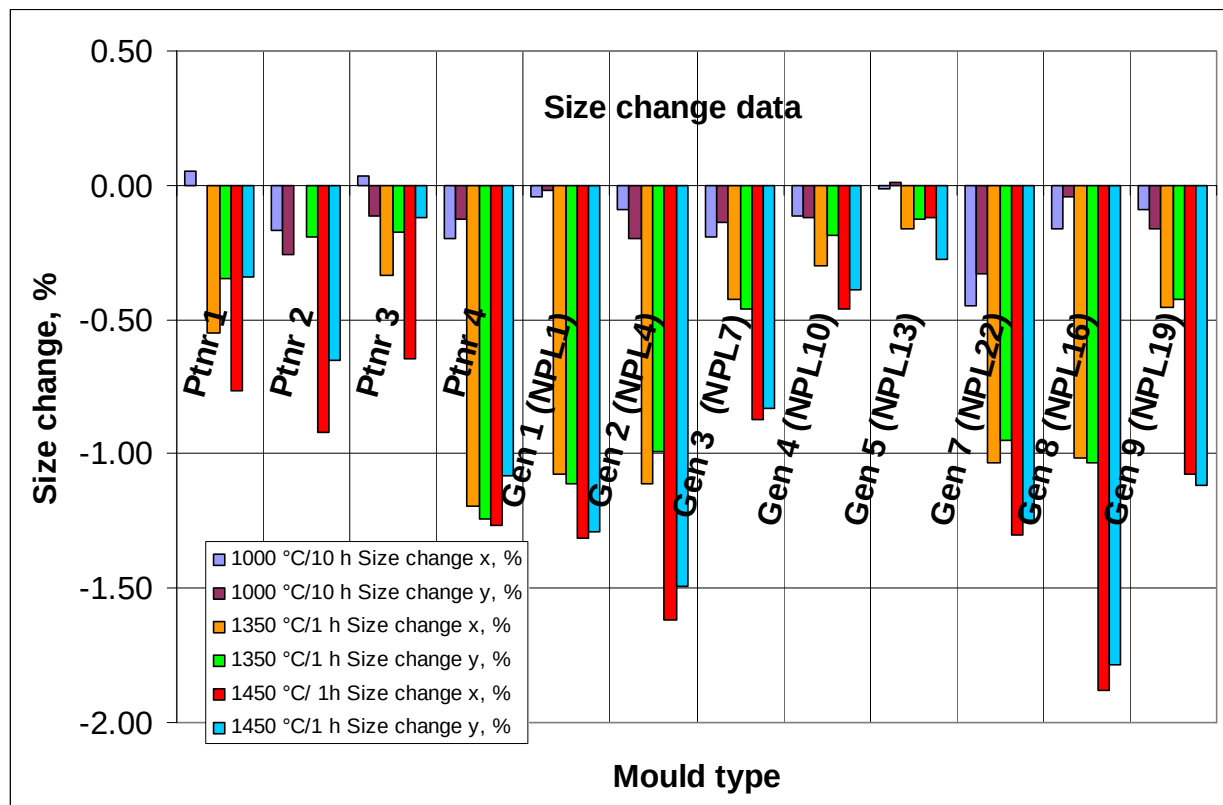


Figure 16: Lateral free dimension changes parallel to (y) and perpendicular to (x) test sample manufacturing vertical direction.

5.4 Flexural distortion

As noted below, there was some indication during flexural creep testing of the development of beam curvature occurring during heat-up in the absence of applied force. The flexural distortion tests were designed to investigate this more systematically. Results are shown in Table 9 and illustrated in Figure 17. Negative values correspond with bowing inwards towards the metal face. Curved test-pieces are shown in Figure 18.

Table 9: Flexural no-load distortion

Material		Radius of curvature, mm			Curvature, mm ⁻¹		
		1000 °C/10h	1350 °C/1 h	1450 °C/1 h	1000 °C/10h	1350 °C/1 h	1450 °C/1 h
Partner 1	PJS	-2373	-1075	-643	-0.000421	-0.000930	-0.001554
Partner 2	PJU	-4003	-838	-633	-0.000250	-0.001193	-0.001580
Partner 3	PJV	177305	-2919	-851	0.000006	-0.000343	-0.001175
Partner 4	PKB	9543	1911	19793	0.000105	0.000523	0.000051
Generic 1	PJX	3216	3650	9740	0.000311	0.000274	0.000103
Generic 2	PJZ	8899	3168	1830	0.000112	0.000316	0.000546
Generic 3	PKF	4029	1196	972	0.000248	0.000836	0.001029
Generic 4	PKJ	-8038	-14590	-6559	-0.000124	-0.000069	-0.000152
Generic 5	PKL	-2581	-16168	-5211	-0.000387	-0.000062	-0.000192
Generic 7	PLA	4163	1511	6842	0.000240	0.000662	0.000146
Generic 8	PKS	25542	11135	31956	0.000039	0.000090	0.000031
Generic 9	PKY	45343	11994	4835	0.000022	0.000083	0.000207

It is interesting that the Partner moulds, with the exception of Partner 4, tended to give negative curvature, i.e. the metal contact side becoming convex while the generic moulds mostly gave positive curvature with the metal contact face becoming concave. It can also be noted that the curvature generally increases with increasing firing temperature, and that most moulds show a tendency to bend even after firing at only 1000 °C, despite the fact that the lateral shrinkage is still small. It should be noted that these results show free, unrestrained curvature of a strip test-piece. In practice, in a three-dimensional shaped mould the effect will be much less because of mutual restraint of the closed geometry of the mould shape. The effect will become most strongly manifest in the casting of flat plates or similar shapes with limited restraint.

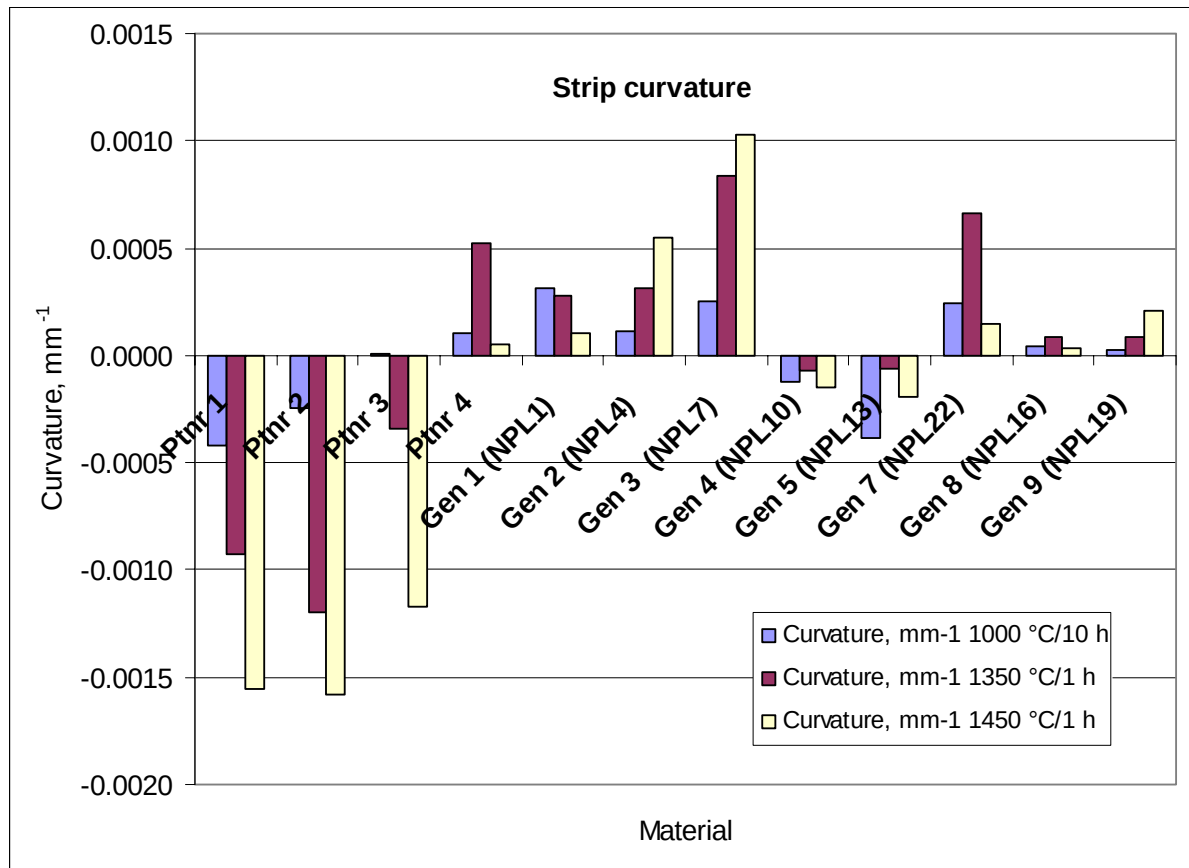
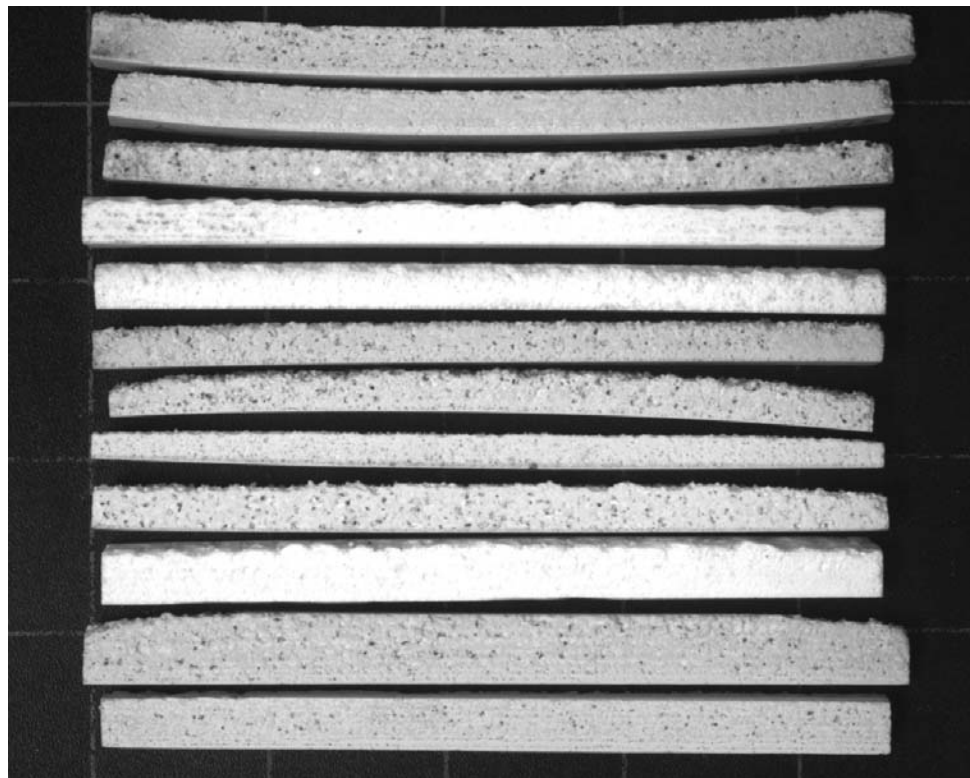


Figure 17: Flexural distortion curvature, all test-pieces cut parallel to length of samples.



Partner 1, PJS
 Partner 2, PJU
 Partner 3, PJS
 Partner 4, PKA
 Generic 1, NPL1
 Generic 2, NPL4
 Generic 3, NPL7
 Generic 4, NPL10
 Generic 5, NPL13
 Generic 7, NPL22
 Generic 8, NPL16
 Generic 9, NPL19

Figure 18: Test-pieces from the flexural distortion test fired to 1450 °C for one hour. The metal contact face is the lower side of each test-piece.

5.5 Thermal expansion

A typical set of thermal expansion curves is shown in Figure 19. Tests were made to three different programmer maximum temperatures which gave test-piece maximum temperatures of 1240 °C, 1340 °C, and 1430 °C. In the case of the Partner 3 material in Figure 19, the heating traces are smooth, consistent and monotonic to about 1000 °C after which there is the onset of permanent shrinkage due to sintering. With increasing maximum temperature there is an increase in the shrinkage resulting from sintering, and a concomitant increase in the magnitude of the small cristobalite transition on cooling below 220 °C. There also appears to be a trend towards increased shrinkage measured parallel to the manufacturing length (vertical direction) compared with perpendicular to this direction.

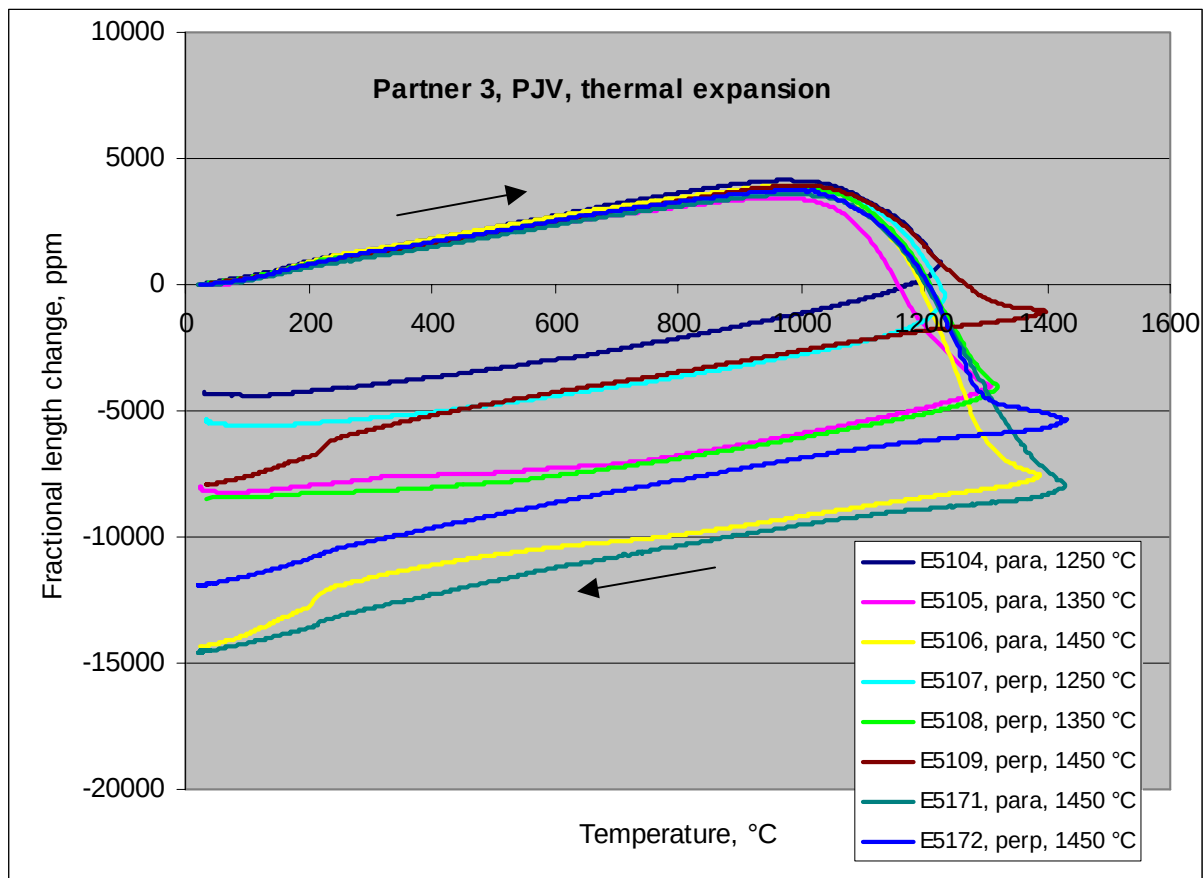


Figure 19: Thermal expansion curves, heating and cooling for single thermal cycles to different maximum temperatures and for test-pieces cut parallel to and perpendicular to the manufacturing length.

The repeatability of the shrinkage is not good in this test, and this is probably a result of two factors: specimen inhomogeneity from small test-piece to small test-piece; and the effects of the pushrod force of about 2 N on the localised contacts at the ends of the test-piece which may cause creep deformation. Nevertheless, the general shape of the curves is consistent, and gives an overall shrinkage of about 1.5% in the parallel direction after heating to 1420 °C, which is rather greater than that measured on freely supported material. It has to be concluded that shrinkage values tend to be exaggerated somewhat in thermal expansion tests.

Figure 20 shows rather different behaviour of the full silica material Generic 1, PJX. The expansion is effectively zero to about 800 °C above which there is a small sintering contraction to the peak cycle temperature. On cooling a low expansion coefficient is maintained to about 600 °C, after which behaviour is dominated by the cristobalite content. There is a clear correlation between the magnitude of the size change due to the beta to alpha cristobalite phase change below 220 °C and the maximum temperature of the cycle. This material also appears to behave more consistently in the two measurement directions than is shown by the Partner 3 material (PJV) shown above.

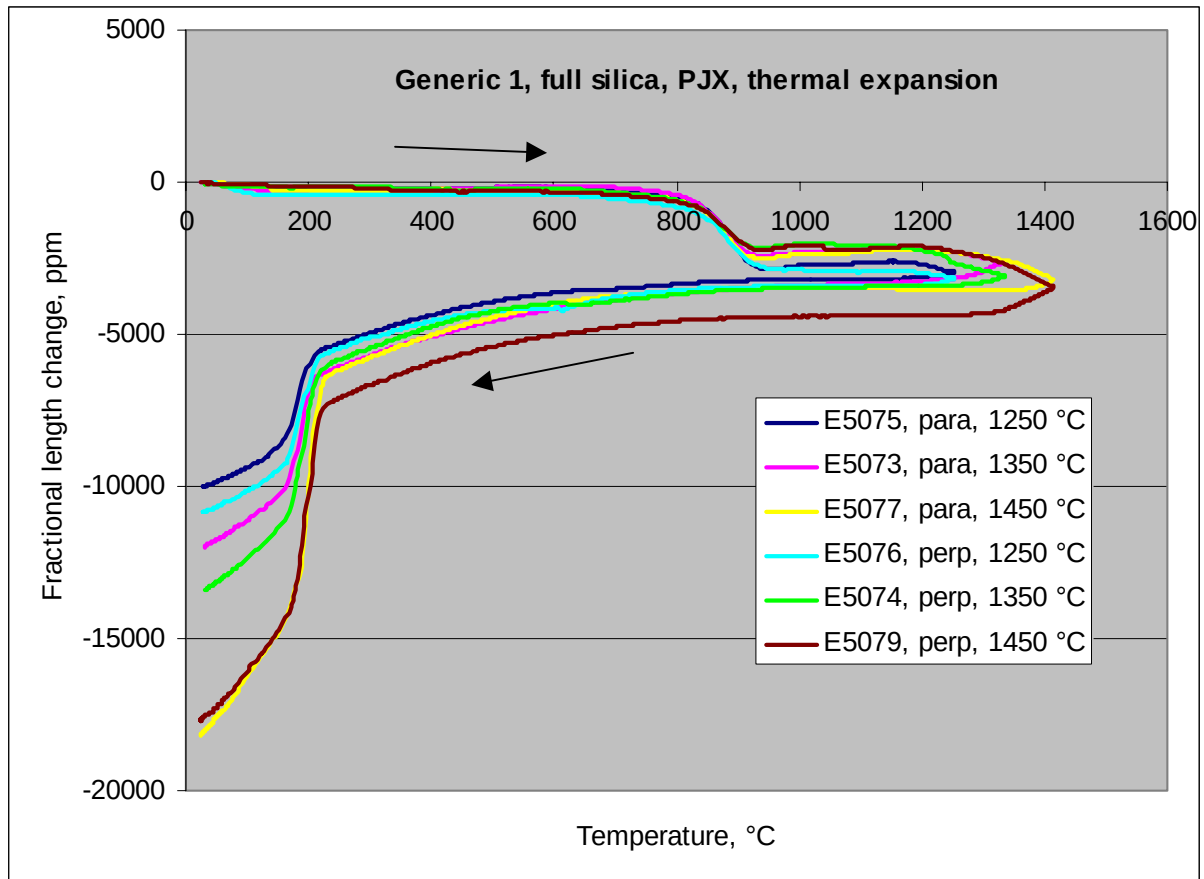


Figure 20: Thermal expansion traces for the full silica material Generic 1 showing low thermal expansion to various maximum temperatures with little hot shrinkage but increasing contributions to cold shrinkage from the cristobalite transformation below 220 °C with increasing maximum temperature.

In order to display comparative data, the following summary data sets have been prepared:

- (1) Mean linear expansion coefficient in the green state from cold to 1000 °C, as a representation of the difference between cold and initial casting dimensions;
- (2) The fractional size change from RT to the peak temperature of the thermal expansion cycles, *i.e.* the size change from RT to a given hot condition incorporating sintering;
- (3) The total size change over a thermal cycle, from RT as a green body back to RT as a fired body, as a measure of the overall size change and whether this will cause any inappropriate mismatch with the cooling metal.

Figure 21 shows the mean linear expansion coefficients. They cover a wide range. Of the generic materials, the full silica system (Generics 1 and 7) has the lowest expansion coefficient, in fact slightly negative to 1000 °C as a consequence of sintering before this temperature is reached. The full aluminosilicate system (Generic 2 and 8) and the zircon/molochite systems (Generic 3 and 9) have intermediate values, while the zircon/alumina system (generics 4 and 5) have high expansion coefficients.

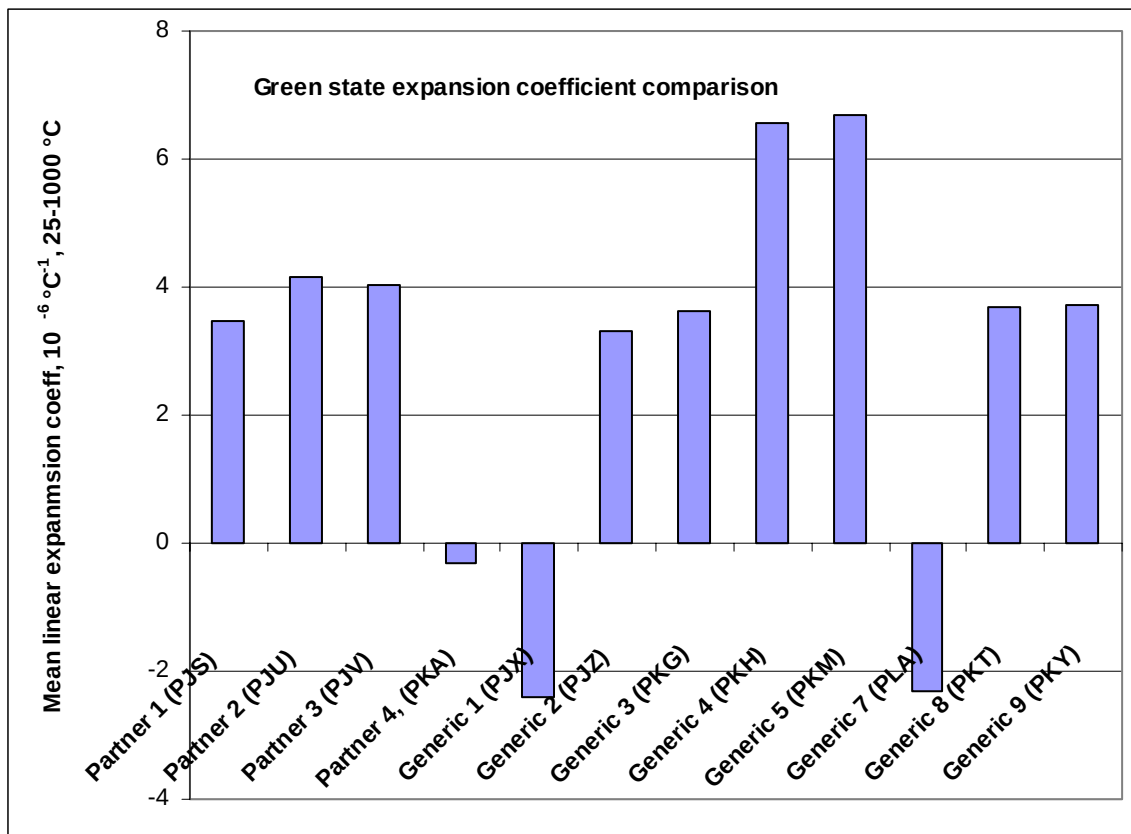


Figure 21: Green state expansion coefficients, RT – 1000 °C.

Size change from RT to the maximum temperature of the cycle is shown in Figure 22(a), and from RT to RT over the cycle is shown in Figure 22(b). The zircon/alumina system (Generic 4 and 5) shows least overall shrinkage, and the full silica (Generic 1 and 7) and the full aluminosilicate (Generic 2 and 8) show the most shrinkage. It is clear that different expansion allowances have to be made for the different material types.

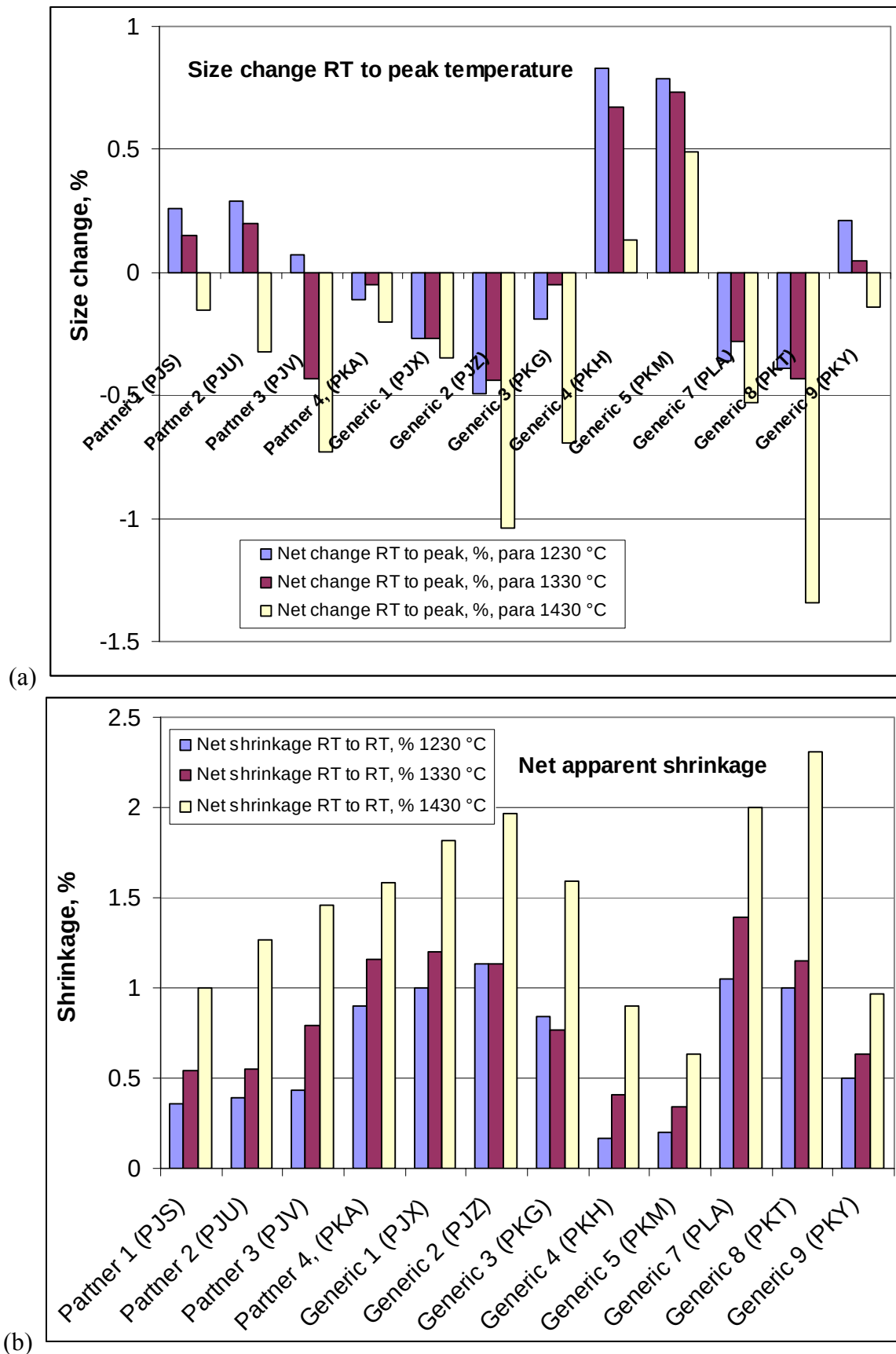


Figure 22: Size changes (a) on heating to the peak temperature of the cycle, and (b) from cold to cold over the cycle.

5.6 Flexural creep

A typical raw data set is plotted in Figure 23(a) for a test-piece of Partner 4 material (PKA) subjected to the standard firing cycle with a maximum of 1350 °C and with the test force applied after two minutes at 1350 °C. There is a distinct creep curve shown in Figure 23(b), although the amount of creep obtained during the short loading period is very small. This example illustrates the sensitivity of the system, and also serves to demonstrate that the amount of creep can be small. Figure 24(a) compares the result in Figure 23(b) with a second test at 1450 °C, which shows a slightly lower creep rate. This indicates that the conventional Arrhenius behaviour normally associated with creep does not occur in these materials owing to structural changes.

Similar tests were made for all the test materials. An example is shown in Figure 24(b) for Partner 3 material, which clearly creeps much faster than Partner 4 material. In order to compare all the data, Figure 25 has been prepared giving the nominal flexural creep strain after 60 s at each of the three temperatures 1250 °C, 1350 °C, and 1450 °C. It has been plotted on a logarithmic scale in order to cover the wide range of creep rates. Some data are missing, either because the tests were not undertaken because the higher temperature creep rates were very small, or because of creep runaway before 60 s had elapsed.

It is immediately apparent that the range of creep rates covers more than two orders of magnitude for the different shell systems. The lowest creep rates were obtained from the full silica systems Generic 1 and Generic 7, and from Partner 4. The highest creep rates were obtained from the full aluminosilicate systems Generic 2 and Generic 8. It is also apparent that the temperature sensitivity of creep rate is very different between the different systems; Some, such as Partner 1 and Partner 2 show little or no effect of temperature on creep rate, suggesting that the microstructure is becoming progressively more resistant to creep as the temperature is raised.

It should be noted that these comparisons are based on single tests. There was insufficient time within the project to undertake multiple testing. However, some confidence can be gained from the similarity of rates determined for the following nominally equivalent pairs of materials: Generic 1 and Generic 7; Generic 2 and Generic 8; Generic 3 and Generic 9; Generic 4 and Generic 5.

It should also be noted that the different systems had different tendencies for cracking to be initiated from the tensile face (stucco side) during creep. This was particularly evident in Partner 1, code PJS (Figure 26), and the thicker moulds Generic 8, code PKT and Generic 9, code PKK. The appearance of cracks will tend to accelerate creep rates because the test-piece is no longer deforming homogeneously, but the deformation is concentrated around the growing crack tip.

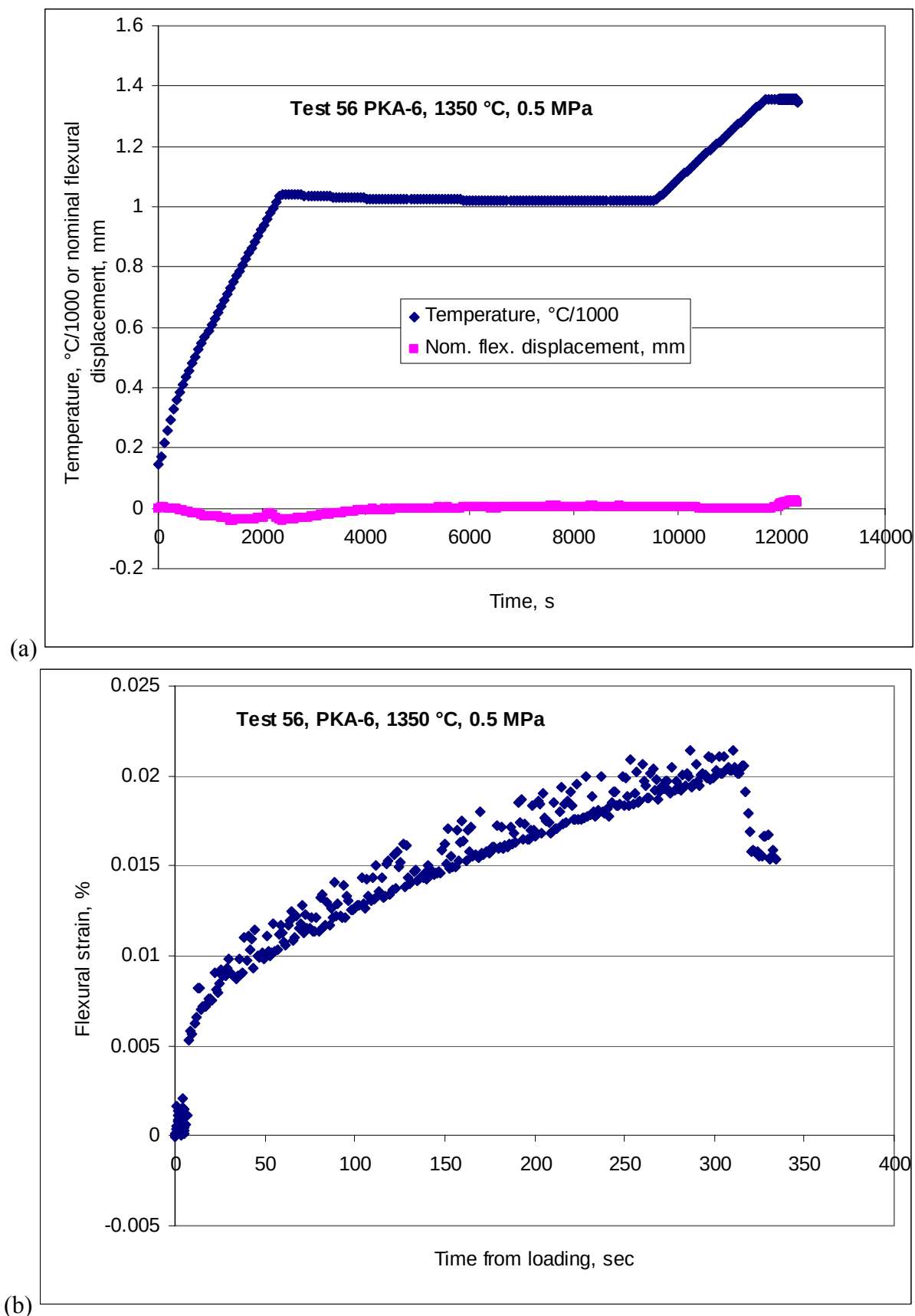


Figure 23: Example of flexural creep raw data for a test on Partner 4 material; (a) temperature and displacement as a function of total elapsed time; (b) nominal flexural strain as a function of time from the start of loading at 1350 °C to unloading after 300 s.

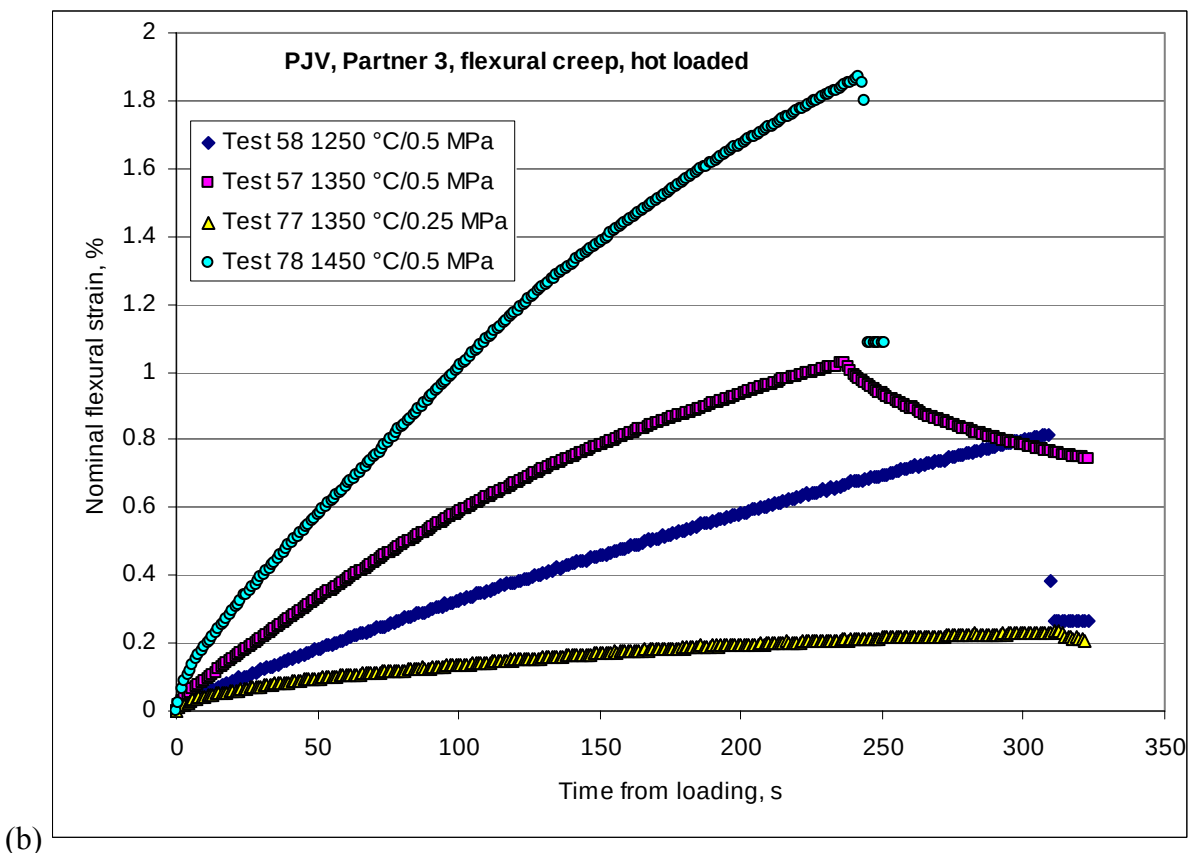
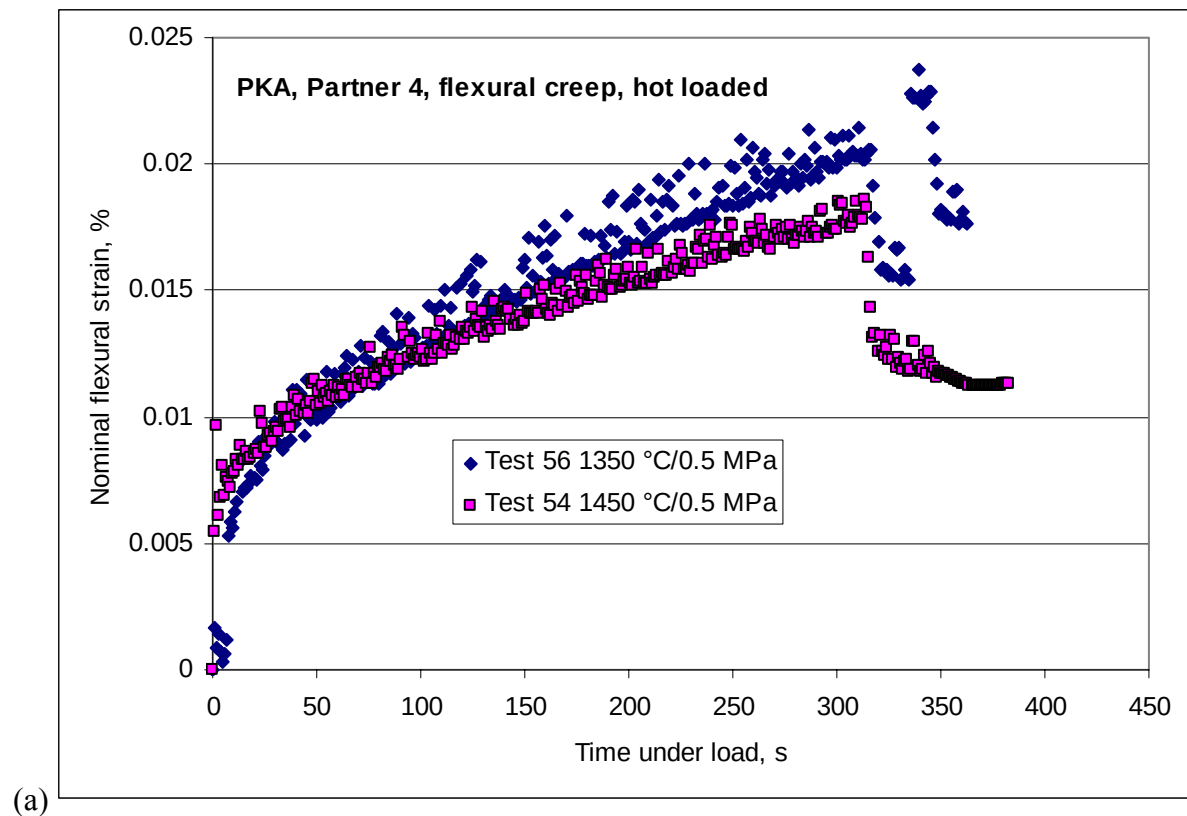


Figure 24: Flexural creep curves on (a) Partner 4 material at 1350 °C and 1450 °C, and (b) Partner 3 material at 1250 °C, 1350 °C and 1450 °C.

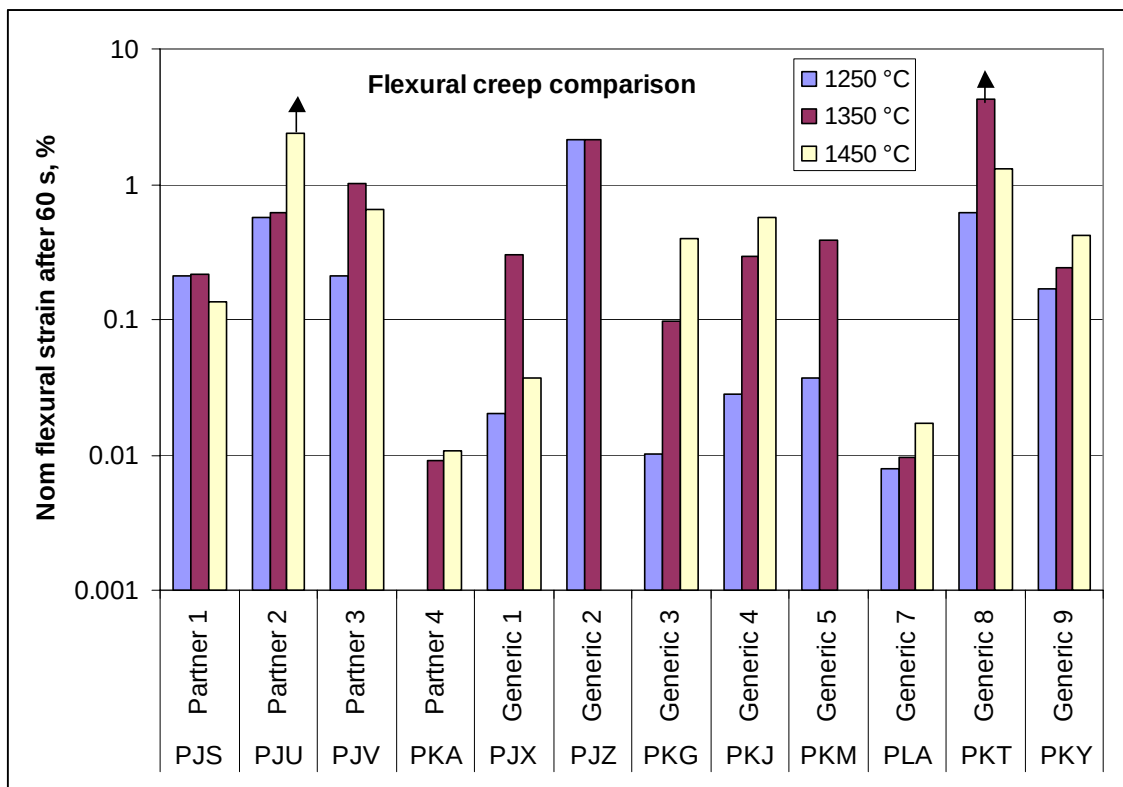


Figure 25: A comparison of nominal flexural creep strain after 60 s at each of the three test temperatures. Note that the 1450 °C result for Partner 2 is for 6 s only, and the result for Generic 8 at 1350 °C is limited by reaching the maximum deflection possible after about 50 s.



Test 60, 1350 °C,
0.5 MPa

Test 61, 1250 °C,
0.5 MPa

Test 80, 1250 °C,
0.5 MPa

Test 81, 1450 °C, no
load, reverse bend

Test 82, 1450 °C
under 0.5 MPa load
from cold

Figure 26: Flexural creep test-pieces of Partner 1, PJS, with test conditions shown at the right. The appearance of cracks is arrowed. Test 81 under no applied force shows reverse bending, while test 82 heated under load from cold shows much greater deformation than any other condition.

It was noted that during the heating stage of the test, the apparent deflection of the test-piece under no load, or strictly, only its own mass counterbalanced by the transducer force, varied significantly from material to material. An example, test 81, is shown in Figure 26. In order to check this aspect out, the beam curvature tests described above were undertaken. In these latter tests, the effects of mass and transducer force could be removed to obtain a truly consistent picture.

Initially, it was decided that the hot-loading of the test-pieces, *i.e.* loading only when they had reached the peak temperature, was the most appropriate way of determining performance. However, it was decided to determine the effect of loading the test-piece before heating, and heating under load to establish whether the dead weight of the mould system, or indeed the effect of molten metal pressure while the mould was heating up, could affect the distortion. Figure 27 shows a typically data set for Partner 4 mould samples, comparing three conditions, loading at 1450 °C, loading at 1000 °C and then heating under load to 1450 °C, and loading from cold to 1450 °C. In those tests in which the test-piece is hot loaded, there is minimal deformation, the total strain detected being less than 0.1%. In contrast, the test-piece that was cold-loaded showed deformation commencing at about 650 °C during heating, leading to accelerating deformation up to about 950 °C after which it declined, with very little further deformation during the hold at 1000 °C and the subsequent heating to 1450 °C. This result indicates that by far the biggest risk of deformation in this material occurs during the pre-heating of the mould system, and not during subsequent casting.

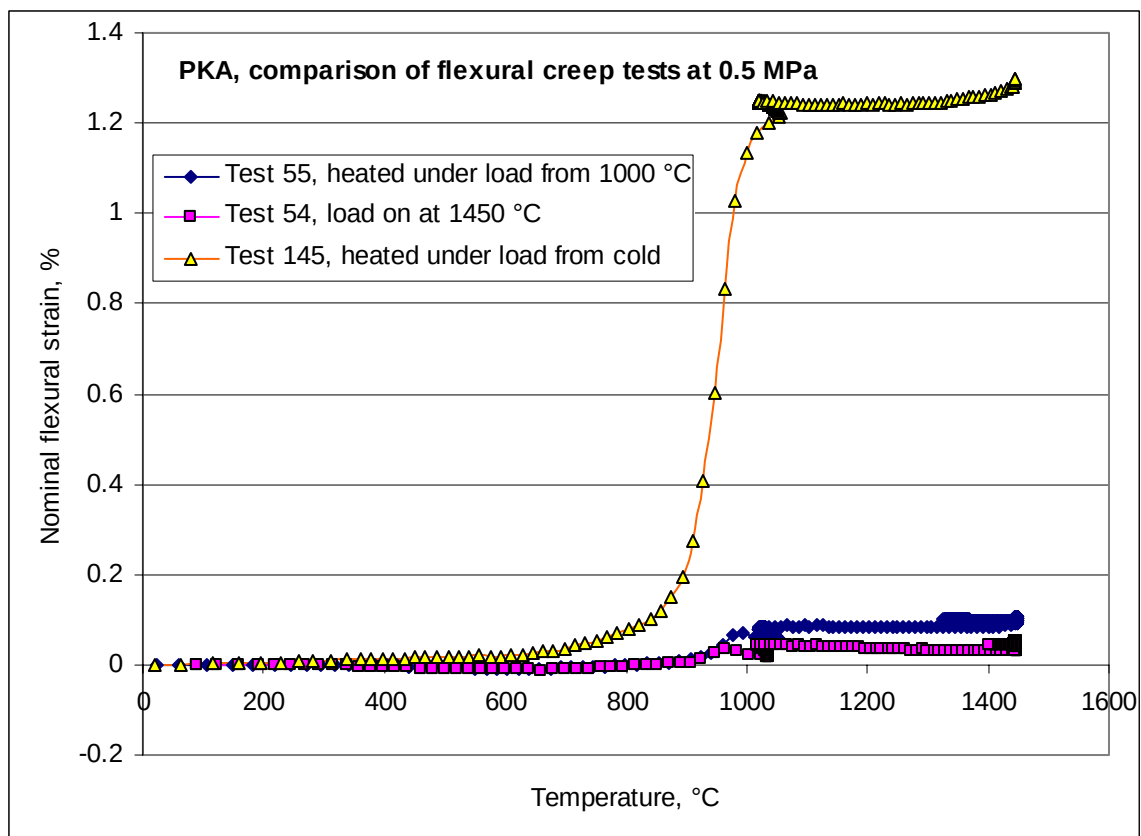


Figure 27: Nominal flexural strain as a function of temperature during heating to 1450 °C with a hold at 1000 °C for 2 h, comparing Partner 4 test-pieces loaded from cold with those loaded at 1000 °C and at 1450 °C.

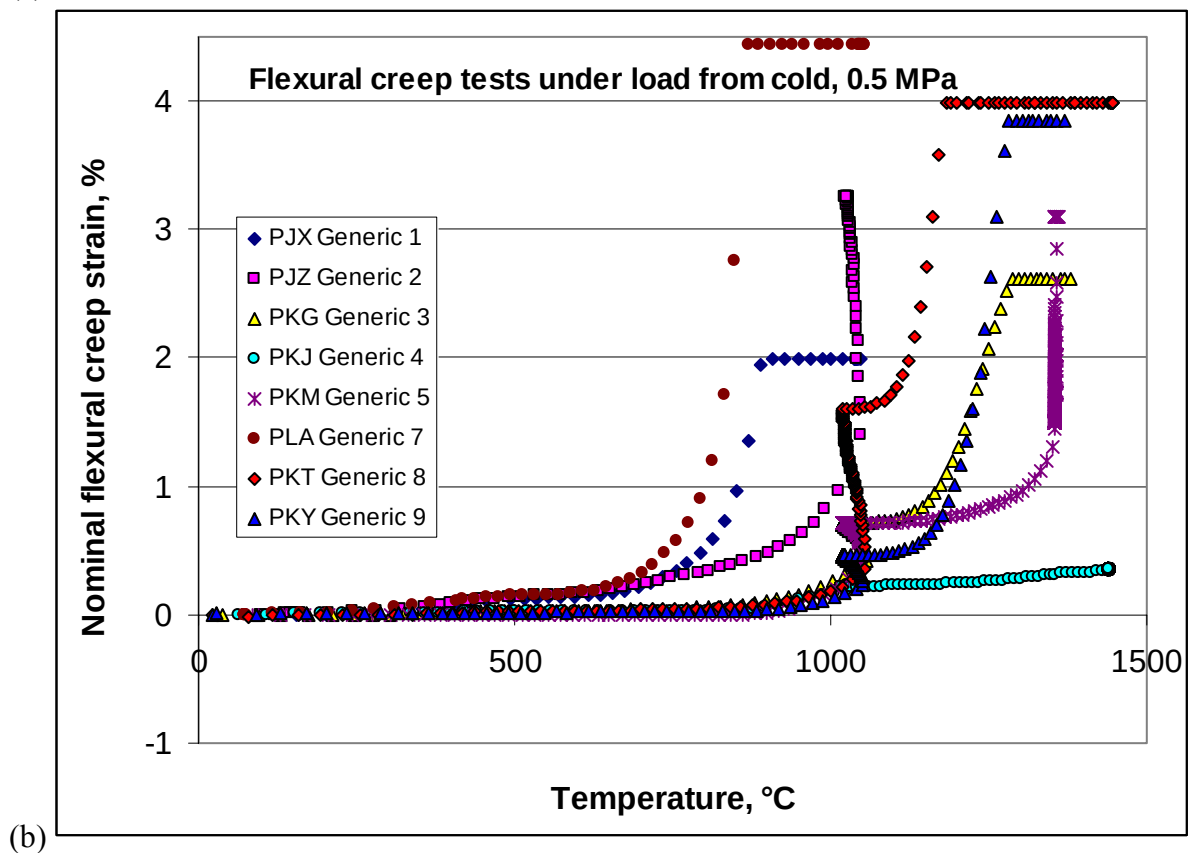
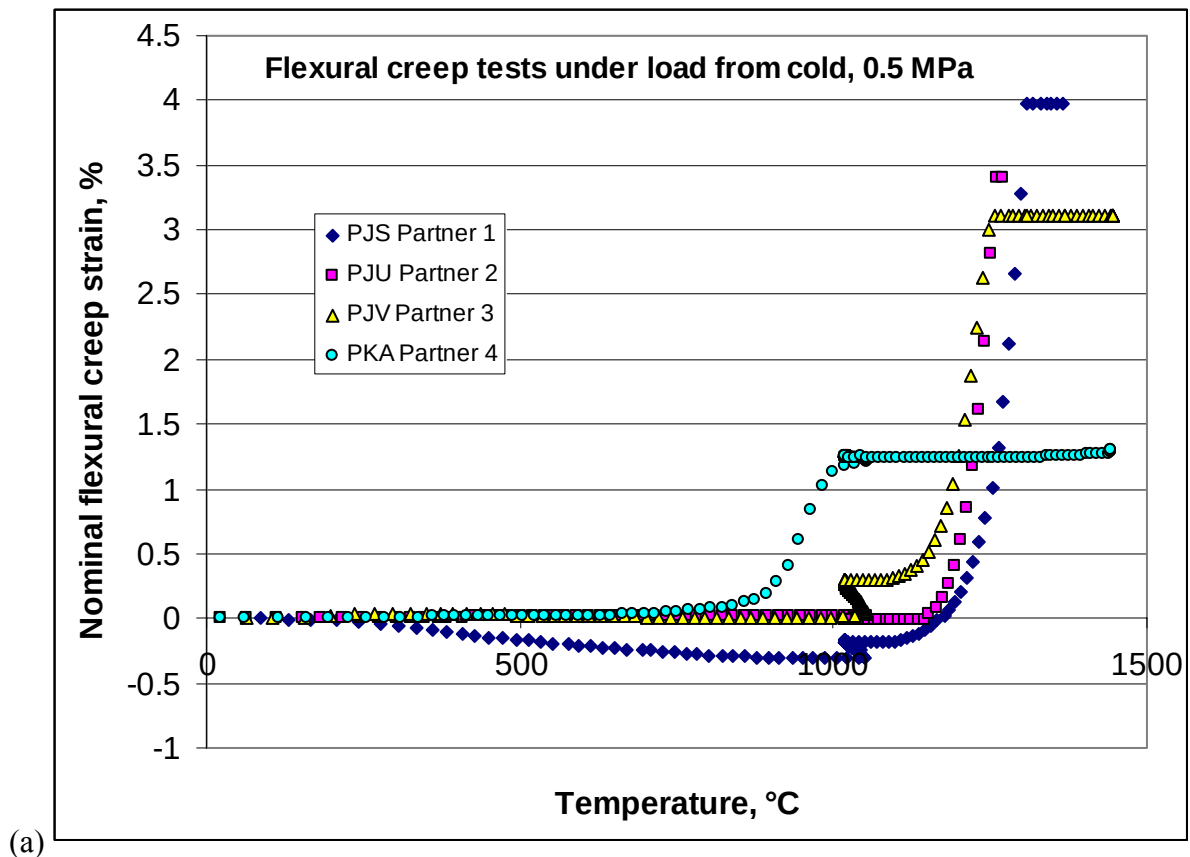


Figure 28: Comparison of flexural creep tests under loading from cold using the same heating cycle of 2 h at 1000 °C and ramp to 1350 °C or 1450 °C. High-temperature horizontal regions of curves denote that the flexural displacement limit of the test jig has been reached. (a) Partner moulds, (b) generic moulds.

Figure 28 shows a comparison of tests on all the materials run under identical heating schedules. The majority of materials are stable to about 1000 °C, but commence deformation above about 1080 °C. The exceptions are: the full silica materials, Generic 1 and Generic 7, which start to deform at about 650 °C; the full aluminosilicate material, Generic 2 and 8, which commences deformation at about 900 °C, but slowly stabilise with continued holding at 1000 °C; and the Partner 4 material, which shows some deformation from about 900 °C but stabilises at 1000 °C, and shows no further deformation to 1450 °C. The most stable material in this test is the zircon/alumina Generic 4 with little deformation up to and including the hold at 1450 °C (note that the similar Generic 5 did not behave equivalently - most of the deformation in this test sample can be attributed to cracking).

It is also noteworthy that the Partner 1 mould, which was found to suffer from significant negative curvature in the curvature tests, also shows this effect in these tests while under load intended to bend it the other direction.

It is thought that the outcome of this type of test complements the hot-loading type of test in respect of understanding better the risks of deformation arising as the mould heats up, compared with that occurring once it is hot and fully sintered.

5.7 Compression creep

Compression creep tests were undertaken and analysed in the same way as for flexural creep. A typical example of the series of hot-loaded creep deformations obtained is shown in Figure 29 for Partner 1 material. In most cases the creep was logarithmic in nature, i.e. progressively slowing down with time. In some cases, mostly with higher creep rates, there was an element of structural collapse and buckling occurring. As can be seen in Figure 29, the two tests conducted at 1450 °C begin similarly, but one plateaus out while the other accelerates to catastrophic failure. Much will depend on the precise local microstructure and the perfection of mechanical alignment.

To check on the alignment issue and on the calibration of the system, the cold-to-cold dimension changes for the first three test series were compared with those recorded during the test. The outcome is shown in Figure 30, in which a reasonable agreement is shown in all except the first two tests.

A comparison of hot-loaded compression tests is shown in Figure 31 for all the materials tested at 1250 °C (*i.e.* excluding the thicker Generic 7, 8 and 9). Again, as with the flexural tests, there is a wide range of creep rates. All the data have been summarised in Figure 32, which shows charts of compression creep strain after 60 s and 120 s of hot loading. As with the flexural testing, the least creep resistant is the full aluminosilicate, Generic 2, while the most creep resistant are the full silica material, Generic 1, and the Partner 4 material.

A few tests were made with loading cold and heating under load. Figure 33 shows the results. As with the equivalent flexural tests, the full silica, Generic 1, shows some movement before 1000 °C is reached, and little thereafter, whereas Generics 3, 4 and 5 show onsets of significant creep above about 1100 °C. The rankings are not the same as for flexural creep, but this may be due to the inhomogeneous nature of the materials and the absence of gross cracking in the case of compression testing.

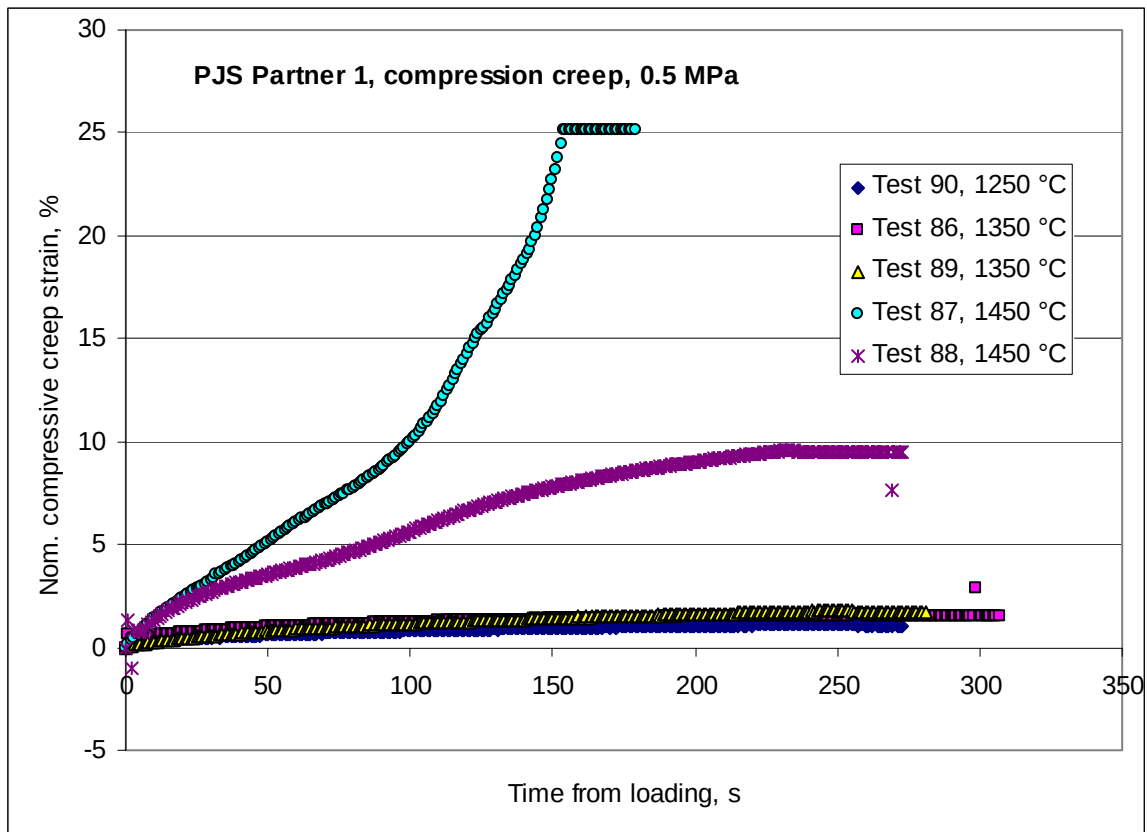


Figure 29: Compression creep curves for hot-loaded Partner 1 material.

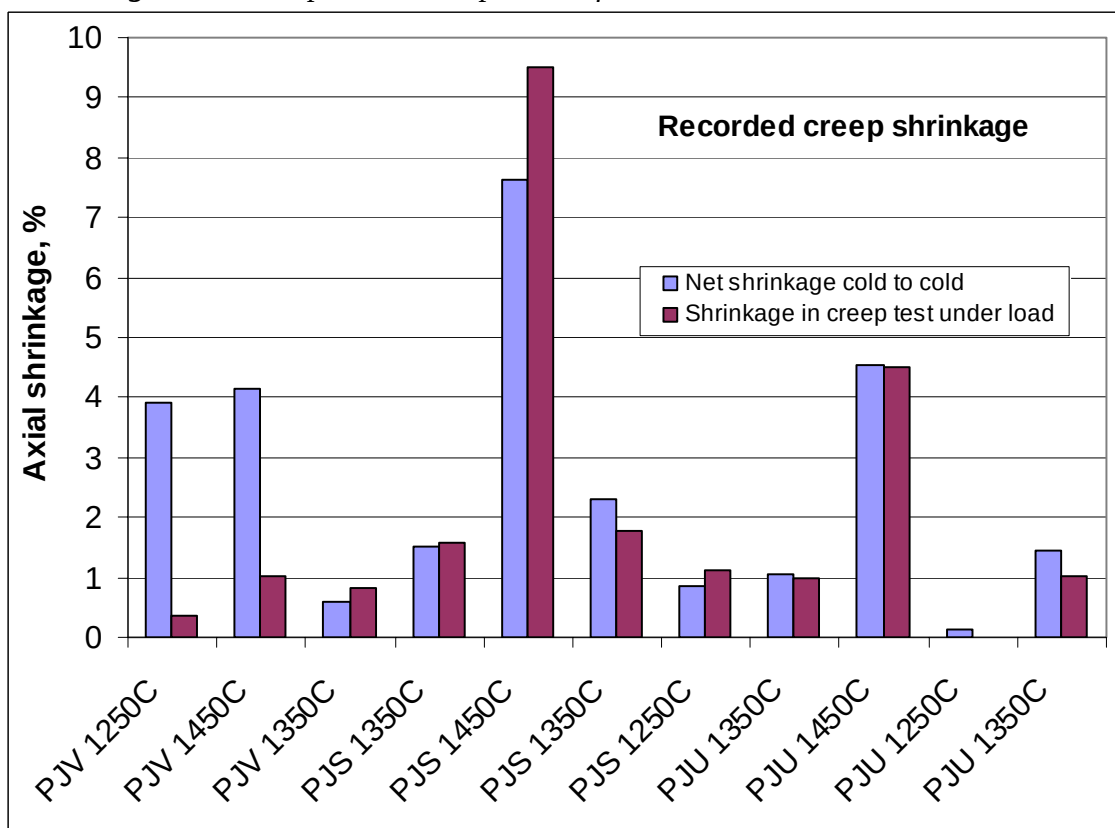


Figure 30: A comparison between cold-to-cold shrinkage and the shrinkage recorded during the test for hot-loaded compression creep tests on three mould materials, Partner 1 (PJS), Partner 2 (PJU) and Partner 3 (PJV).

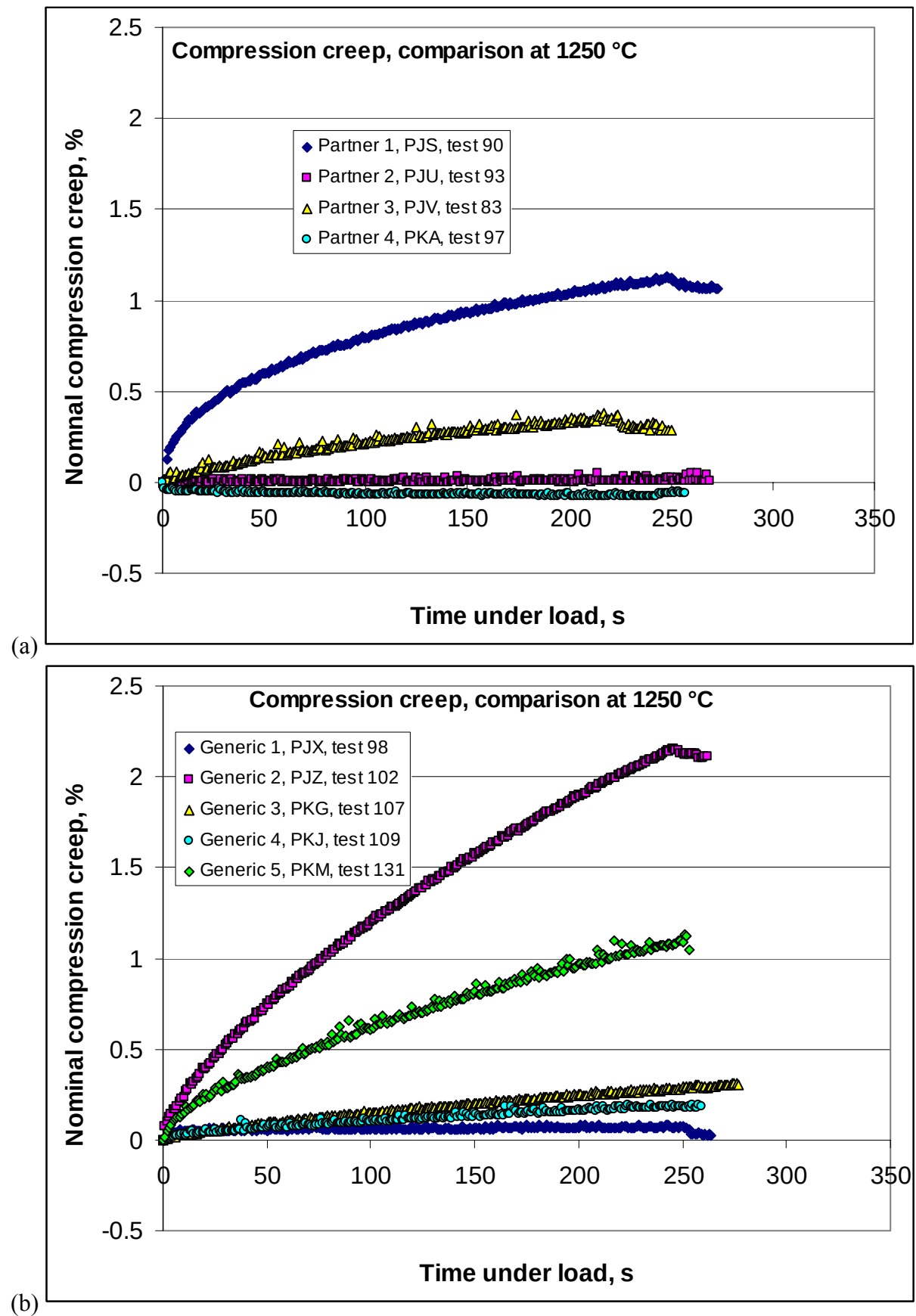


Figure 31: Comparison of compression creep rates for hot loading at 1250 °C.

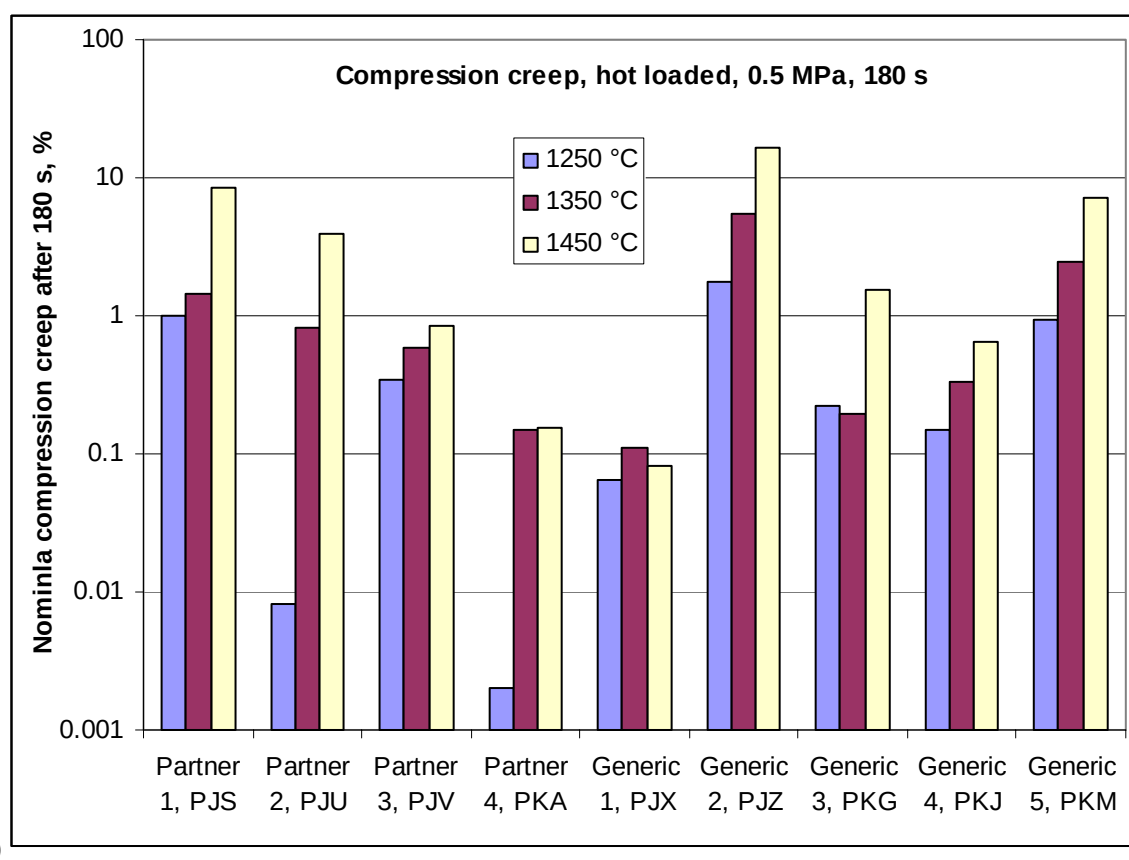
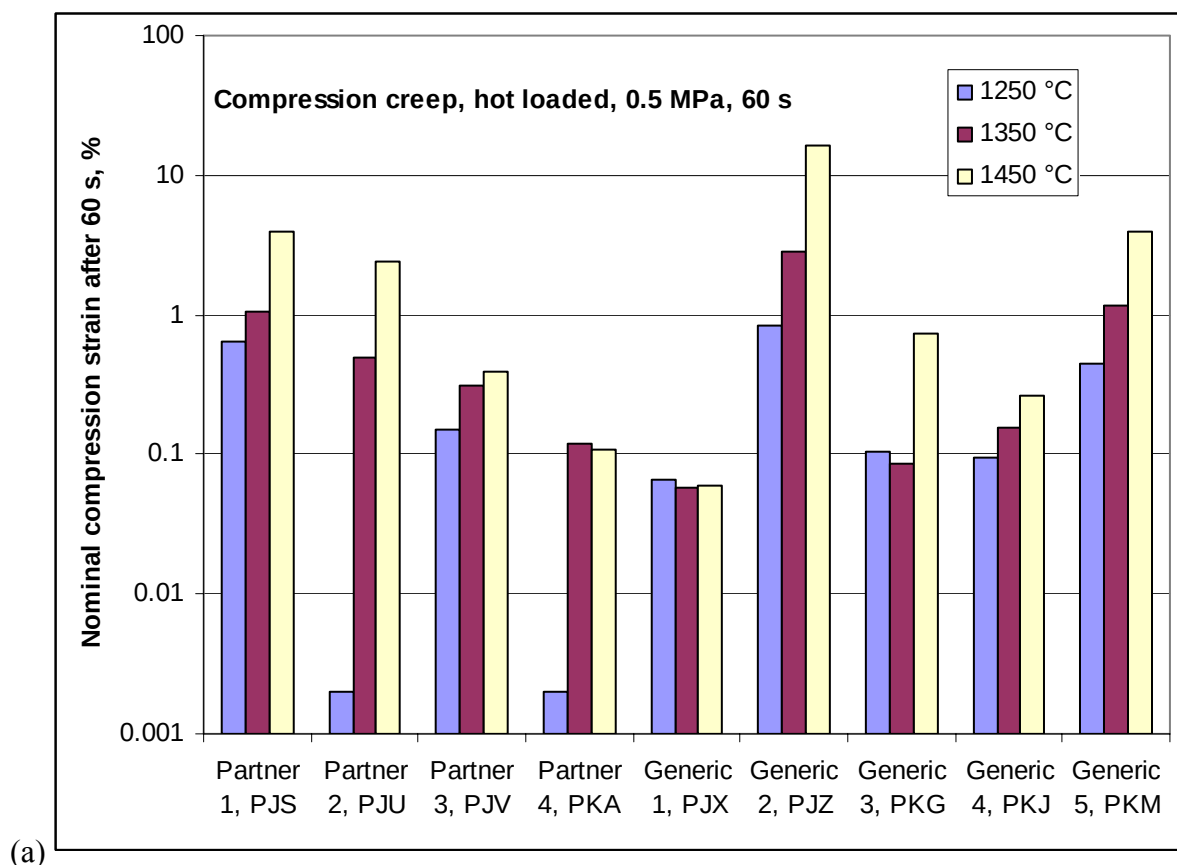


Figure 32: Compression creep strains after (a) 60 s and (b) 180 s of hot loading for all tested materials.

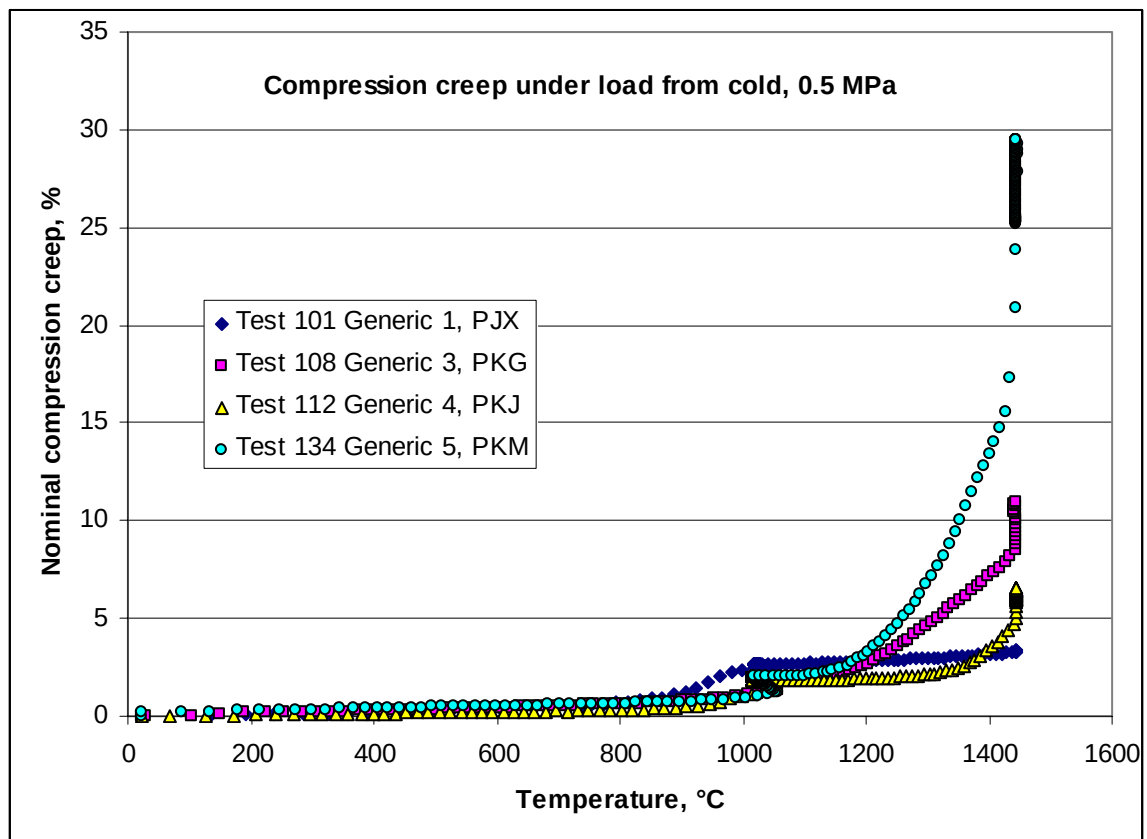


Figure 33: Compression creep tests under load from cold for four generic materials. Test 134 on PKM underwent buckling failure rather than homogeneous creep.

6. CASTING TRIALS

6.1 Mould manufacture and repair

A total of eight shell systems (Nos. 1-5, 7-9) were prepared in duplicate, making 16 shells in total, with dimensional measurements made of the wax thickness as described earlier in 4.1.4. Each shell mould comprised two runners, large and small, each with two waxes of 7.5 mm and 13 mm nominal thickness. Each plate section required a bridge at the lower end to provide mechanical stability. These moulds were delivered to Rolls-Royce after dewaxing, firing for 240 min at 1050 °C and weighing. The weights of the systems are summarised in Figure 34.

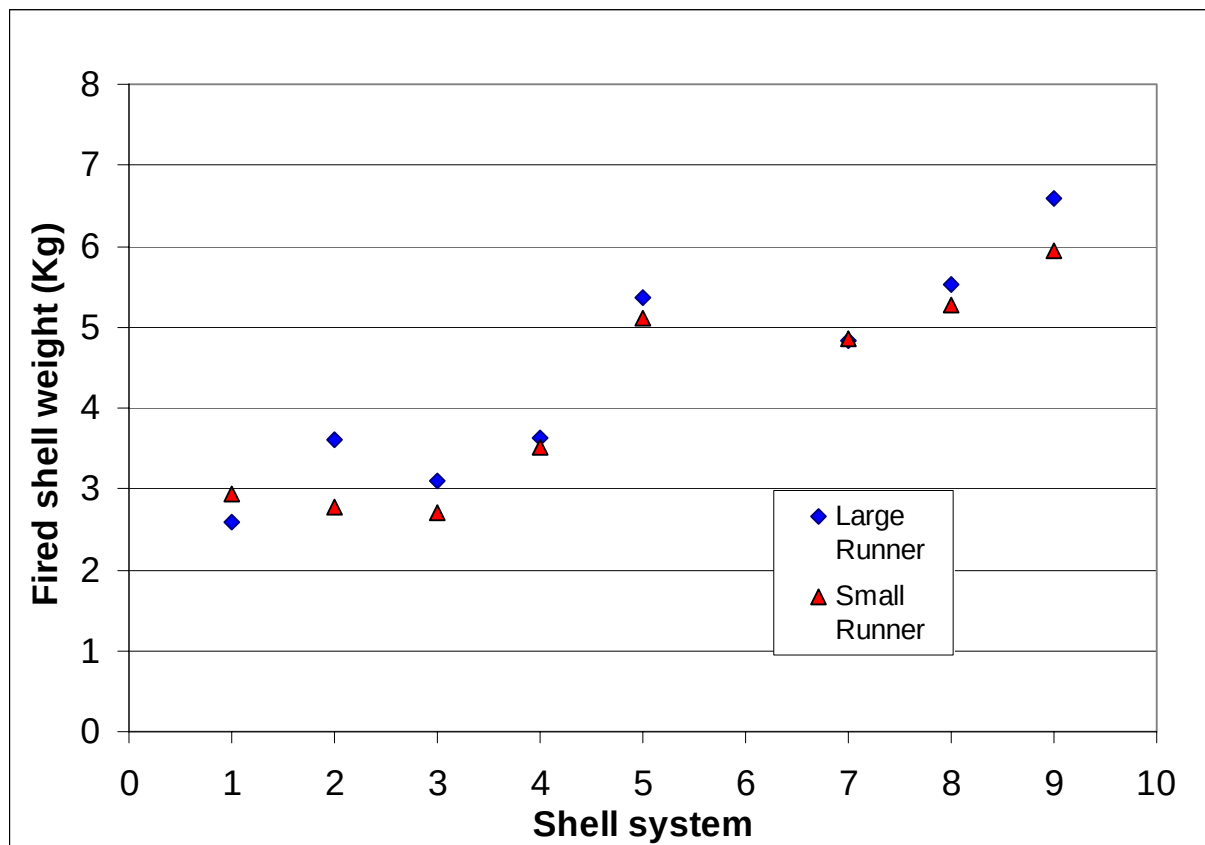


Figure 34: *Weights of the flat plate shell systems prior to casting.*

At Rolls-Royce the moulds were crack tested, and where necessary, repaired by patching (e.g. Figure 35). They were then wrapped with 6 mm of Kaowool ceramic fibre insulation and preheated 1050 °C for a nominal 4 hours before casting.

In addition, four of the industrial Partners were sent similar waxes for building into moulds, some of which required repairing after damage in transit. Of these four Partners, three made mould systems, subjected them to casting and returned the metal plates for dimensional measurement.



Figure 35: Manufactured casting mould and the repair necessary to damage caused either in dewaxing or transit. Note the stabilising bridges between the pair s of waxes.

6.2 Casting

The nickel-based alloy was grade C1023 cast in vacuum at 1500 °C. The alloy composition (%) was:

C 0.16, Cr 15.5, Co 10, Mo 8.5, Ti 3.6, Al 4.2, B 0.006.

The liquidus temperature was approximately 1350 °C.

After casting the moulds were allowed to cool and were then broken open to retrieve the metal plates which were returned to UoB for dimensional analysis.

A summary of the Rolls-Royce casting trials appears in Table 10. In Table 11, the outcome is compared with predictions based on the acceptability of mould quality through mould strength determinations described above. It can be seen that success in obtaining complete castings is clearly linked with the strength of the mould and its resistance to cracking, which is related to thickness and fracture stress.

6.3 Casting dimension analysis – UoB shells

A very large quantity of data was produced in these trials, so the analysis has been restricted to a single line indicated in Figure 36. Annex 3 gives the detailed results from which it is clear that a significant degree of bulging can occur in all systems except the full silica one. This system is the one that is most stable in creep after firing at 1000 °C.

Table 10: Detailed plate casting test outcomes

Assembly no.	Runner size	Shell system*	Remarks	Cast results		
				Full	Partial	Empty
NPL01	L	1 (S1)	Partial metal run-out after pour No metallosstatic head Very variable shell build surfaces No evidence of bulge or creep No evidence of ceramic wetting Rough casting surface	2	0	2
NPL02	S	1 (S1)	Partial metal run-out after pour No metallosstatic head Uneven shell build, thinner on inner plate surfaces No evidence of bulge or creep No evidence of ceramic wetting Very weak shell when cast and cooled Rough casting surface	2	0	2
NPL03	L	2 (AS1)	Full metal run-out after pour Evidence of shell bulge (inner plate surface) Ceramic wetted extensively Uneven shell build on the inner plate surfaces.	0	2	2
NPL04	S	2 (AS1)	Full metal run-out after pour Evidence of shell bulge (inner plate surface) Ceramic wetted extensively Uneven shell build on the inner plate surfaces.	0	4	0
NPL07	L	3 (ZM1)	Full metal run-out after pour Evidence of shell bulge (inner plate surface) Ceramic wetted extensively	0	0	4
NPL08	S	3 (ZM1)	Full metal run-out after pour Evidence of shell bulge (inner plate surface) Ceramic wetted at bottom	0	0	4
NPL17	L	4 (ZA1)	Plates cracked Full metal run-out after pour Evidence of shell bulge before the shell was filled Ceramic wetted by metal	0	0	4
NPL18	S	4 (ZA1)	Plates cracked Full metal run-out after pour Evidence of shell bulge (inner plate surface) Ceramic wetted by metal	0	0	4
NPL15	L	5 (ZA2)	Full shell, no run-out	4	0	0
NPL16	S	5 (ZA2)	Partial metal run-out after pour No metallosstatic head	0	0	4
NPL23	L	7 (S2)	No cracks detected Porous shell Full casting Rough casting surface	4	0	0
NPL24	S	7 (S2)	No cracks detected Porous shell Full casting Rough casting surface	4	0	0
NPL21	L	8 (AS2)	Back right plate cracked All other edges cracked Full casting	4	0	0
NPL22	S	8 (AS2)	Front left plate cracked Based cracked Full casting	4	0	0
NPL13	L	9 (ZM2)	Full casting, plates OK Base cracked	4	0	0
NPL14	S	9 (ZM2)	Front left plate cracked Base cracked Full casting	4	0	0

* details in () are mould types S = silica, AS = aluminosilicate, ZM = zircon/mullite, ZA = zircon/alumina

Table 11: Casting success predictions versus experimental outcomes

Shell system	Description	Casting probability	Result
1	Full silica 1	No	Partial
7	Full silica 2	Yes	Yes
2	Full aluminosilicate 1	No	Partial
8	Full aluminosilicate 2	Yes	Yes
3	Zircon/molochite 1	No	No
9	Zircon/molochite 2	Yes	Yes
4	Zircon/alumina 1	No	No
5	Zircon/alumina 2	Yes	Partial

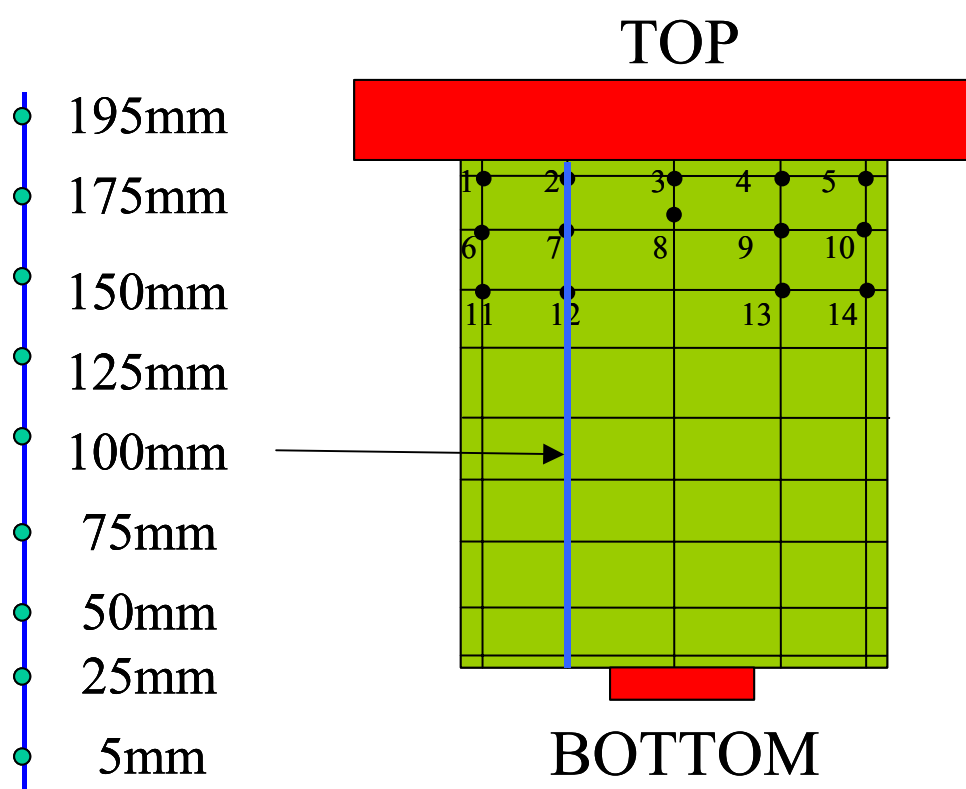
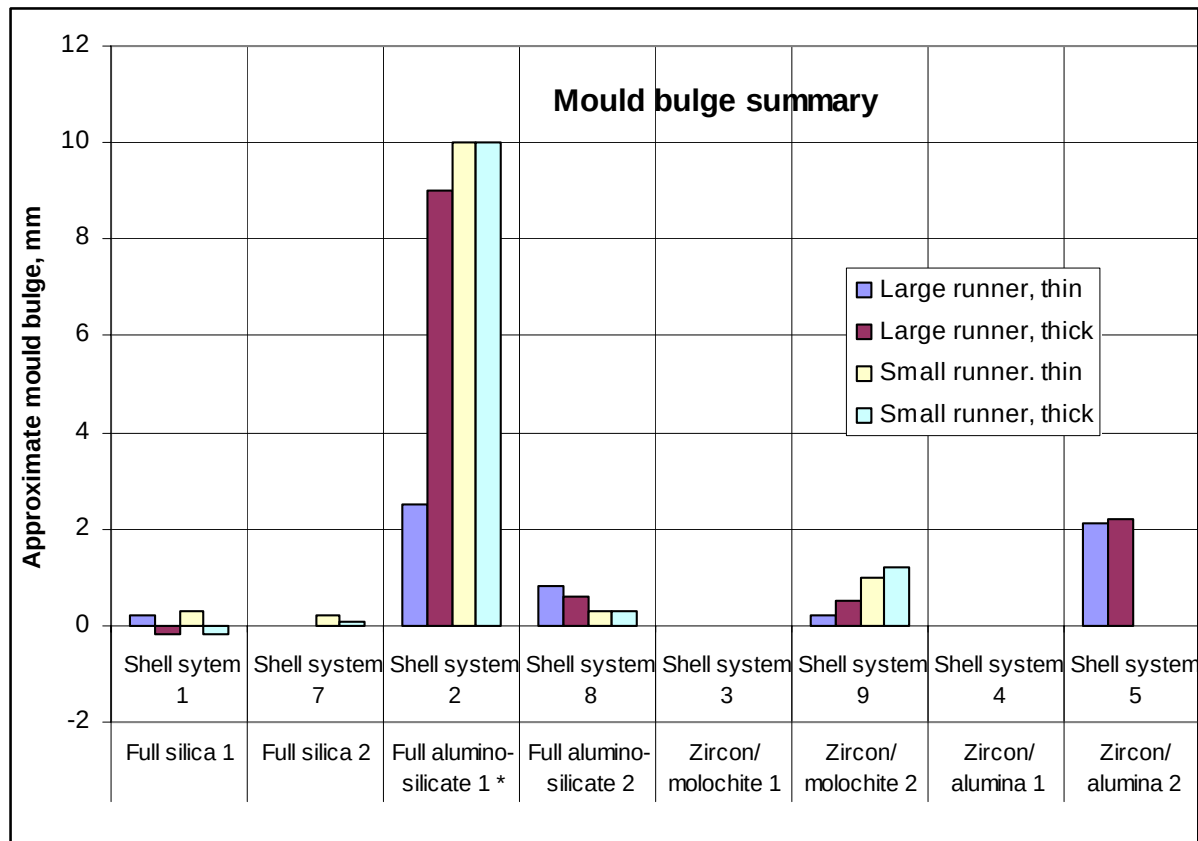


Figure 36: Analysis of data made along the indicated line at the indicated positions on the wax and casting.



* Based on available mould shape data – see Annex 3

Figure 37: Summary of approximate plate bulge results in terms of deviation from the wax dimensions. The bulge is taken at the centre except for the incompletely cast full aluminosilicate 1 shells where it is the maximum measurable.

Figure 37 shows the deviation of the central plate dimensions from the original wax dimensions. It is clear that there is a significant difference between the large and small runner systems. Overall it can be concluded that:

Good Systems:

- Full silica 2 (primary)
- Full silica 1 (primary)
- Full aluminosilicate 2
- Zircon/molochite 2
- Zircon/alumina 2 (small)

Bad Systems:

- Full aluminosilicate 1
- Zircon/molochite 1
- Zircon/alumina 1

Generally the larger runner system provided for less bulge than the small runner system

6.4 Casting dimension analysis – Partner moulds

Equivalent data for moulds prepared by the Partners, based on supplied waxes and subjected to casting tests in their own facilities, are also given in Annex 3, and a summary appears in Figure 38.

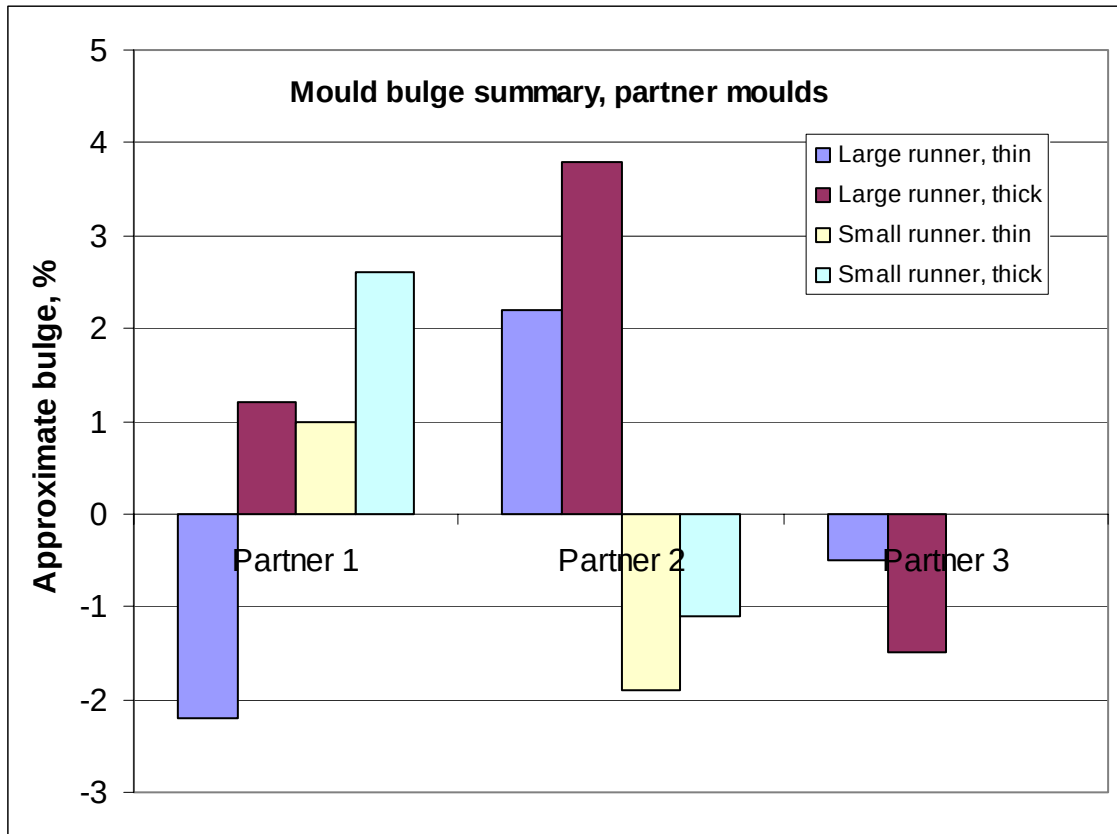


Figure 38: Mould bulge summary for Partner moulds

7. DISCUSSION

Ceramic shell mould systems are complex structures which change properties as they are heated during the metal casting operation. Consequently, property data acquisition and interpretation is rather difficult. This project has shown that there are clear differences in behaviour between the different generic and Partner casting mould systems investigated. Using techniques of flexural and compression creep testing with load application at high temperature and during heating, it has been shown that different systems have different degrees of vulnerability in different temperature ranges. This would seem to correlate with the temperature regimes in which the microstructure is particularly weak. For the silica system this regime is below 1000 °C, above which the microstructure consolidates and shows very little distortion under subsequently applied load. For the aluminosilicate and alumina-based systems it is in the region just above 1000 °C. It is therefore important to consider the deformations possible in the shell moulds before casting takes place and to minimise these by avoiding, particularly, high bending stresses. The project has also shown that mould materials in which deformation can be readily induced in laboratory testing are also prone to significant distortion during casting using the plate bulge test.

In view of the difficulties in mimicking in laboratory tests the transient and variable behaviour in metal casting, it is considered that the data obtained in this project can be used in the first instance primarily in a comparative sense. Heating rates during the casting operation must be extremely high, and steep through-shell temperature gradients must be produced transiently. Neither of these factors can be adequately modelled in laboratory tests. Additional factors in real moulds are the mould wall thickness in relation to the thickness of the original wax former and to the curvature of edges. Thicker moulds provide more mechanical support to resist dimensional changes, but a larger heat sink for the molten metal, affecting the quality of castings. Creating the balance for a given mould material is still likely to be governed by experience rather than modelling.

Acknowledgements

- To David Ford of DAF Associates for masterminding the project direction and coordinating the industrial involvement and the casting trials.
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- To Partner Minco for provision of particle sizing information.
- To the Cast Metal Federation for provision of facilities for meetings and a financial contribution.
- To Rolls-Royce plc, particularly to Dave Evans and Dennis Rogers, for provision of casting facilities for the plate bulge test.

References

1. Chapman, L, Morrell, R, Quested, P.N., Brooks, R.F., Brown, P., Chen, Li-Hung, Olive, S., Ford, D.A., "PAMRIC – properties of alloys and moulds relevant to investment casting" NPL report MATC (D) 141, March 2003.
2. Metals Reference Book, Volume 15, p204, ASM International, USA.
3. Gebelin, J.-C., Jones, S., Jolly, M. R., "Modelling of the De-waxing of Investment Cast Shells", Computational Modelling of Materials, Minerals and Metals Processing, San Diego, USA, September 2001.
4. (a) BS 1902 Methods of test of refractory materials, Part 10. Investment casting shell mould systems, Section 10.1, Determination of resistance to deformation at elevated temperatures (1993); (b) *ibid*, Section 10.2 Determination of permeability and standard air flow capacity at elevated temperatures (1994).
5. NEL reports: (a) Lang, W., Process Research Core-Shell Technology Programme, report to BICTA R&D Seminar, 20 January 1988; (b) Anon., Process Research Core-Shell Technology Programme, report to BICTA, undated (1990?); (c) McCallum, R., More fundamental aspects of investment shell moulds and modified molochite trials, report 913/1, NEL, March 1991.

ANNEX 1: STRESS CALCULATIONS FOR MOULD WALLS UNDER PRESSURE

The stress in the mould wall during casting resulting from the pressure of the molten metal can be estimated for given geometries of mould. This procedure was used to select a relevant stress level for undertaking the compressive and flexural creep tests at high temperature.

The typical pressure at the bottom of a mould system is that due to a maximum of, say, a 1 m head of liquid metal, which exerts a pressure of 0.075 MPa assuming a density of 7500 kg/m³.

The stress at a diameter d in the wall of a tube of inside diameter d_1 and outside diameter d_2 as a result of an internal pressure p is given by⁵:

$$\sigma_h = \frac{(d_2^2 + d^2)d_1^2}{(d_2^2 - d_1^2)d^2} p \quad (\text{A1.1})$$

which is a maximum in the hoop direction at the inner wall, $d = d_1$. Figure A1.1 shows the maximum stress as a function of mould wall thickness for three mould bore diameters.

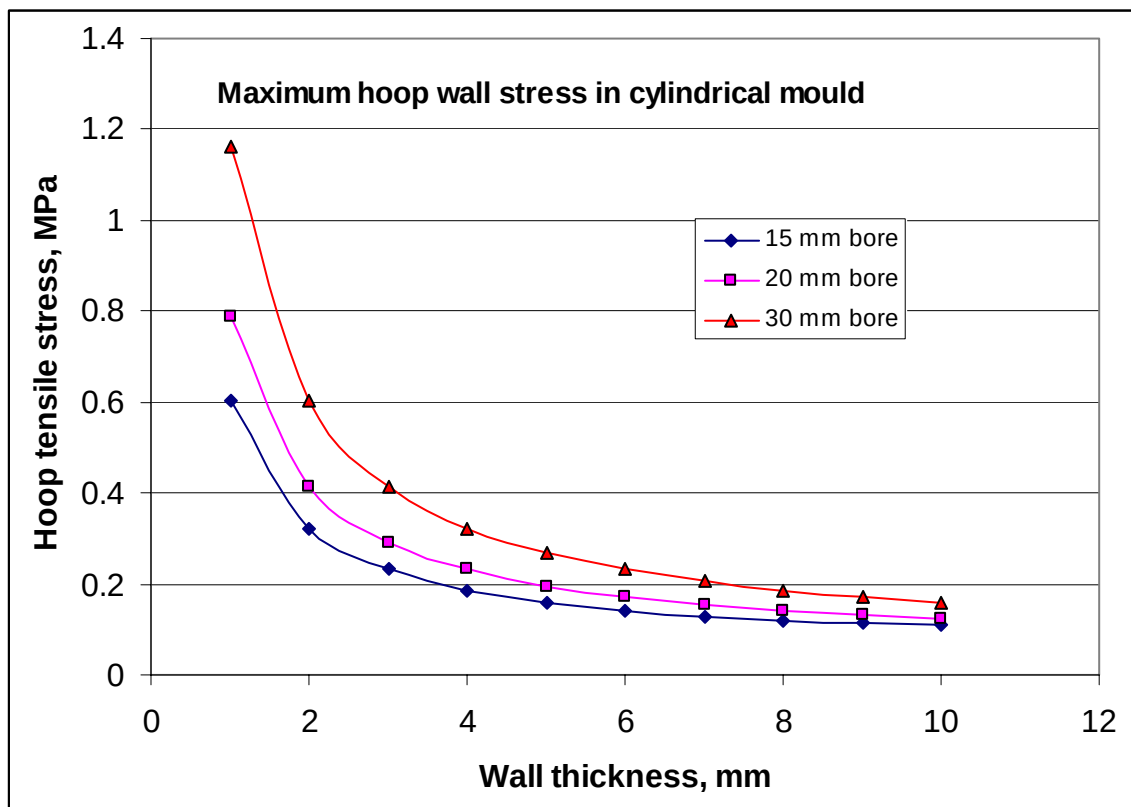


Figure A1.1: Cylindrical mould-wall hoop stress for an internal metallosstatic pressure of 0.075 MPa.

⁵ Ryder, G.H., *Strength of materials*, MacMillan and Co. Ltd, London, third edition, 1965, p 270.

A similar calculation can be made for flat discs. In this case it is necessary to choose appropriate edge conditions, and it is thought that the most relevant are so-called ‘clamped’ or ‘built-in’ edges. In this case the maximum radial stress in the disc of radius a under uniform pressure p is given by ⁶:

$$\sigma_{\max, \text{edge}} = \frac{3pa^2}{4h^2} \quad (\text{A1.2})$$

where h is the disc thickness. For a metallostatic pressure of 0.075 MPa, Figure A1.2 shows the variation in maximum tensile stress as a function of disc thickness for four different diameters.

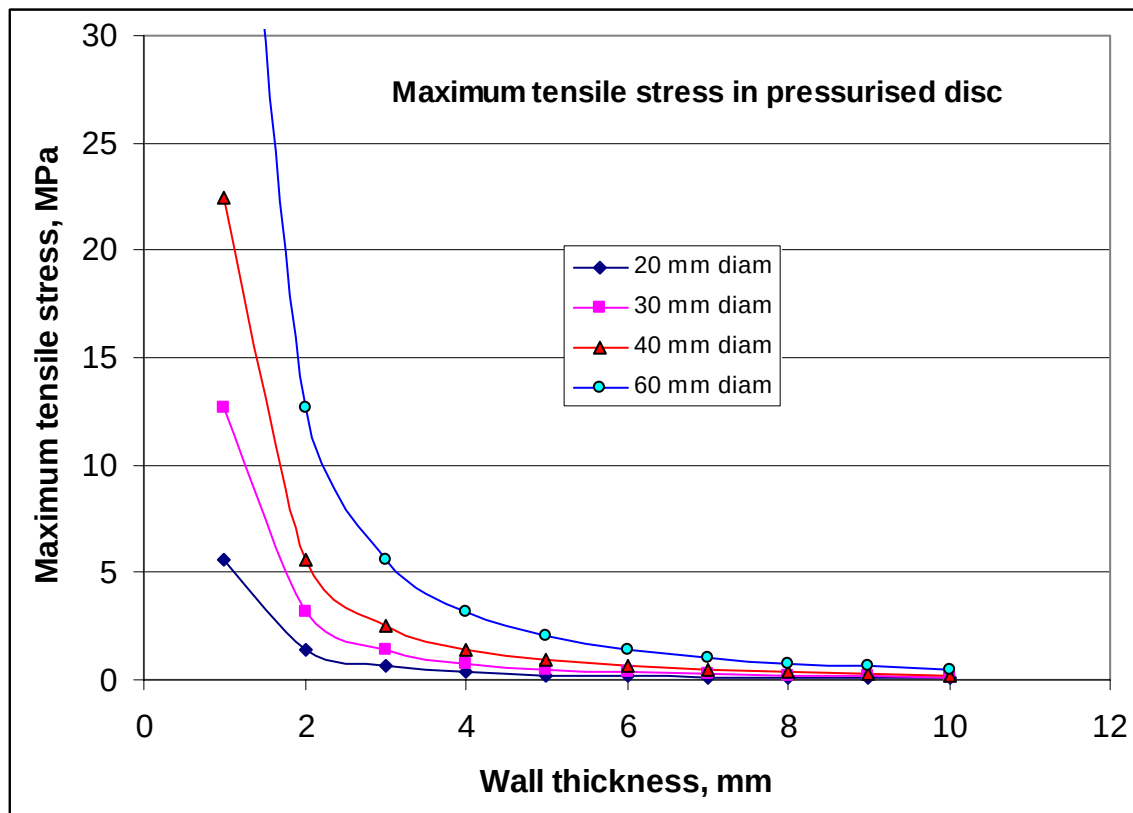


Figure A1.2: Maximum radial stress in disc with ‘built-in’ edge as a function of wall thickness resulting from a metallostatic pressure of 0.075 MPa.

It is clear from these two calculations that the stress levels are highly geometry dependent, and that a flat region in a mould is much more susceptible to damage than a tubular region. On the basis of these calculations, and taking into account that mould wall thicknesses are seldom less than 4 mm and can be adjusted to suit the cast shape, a stress level of 0.5 MPa was chosen as being representative of severe conditions.

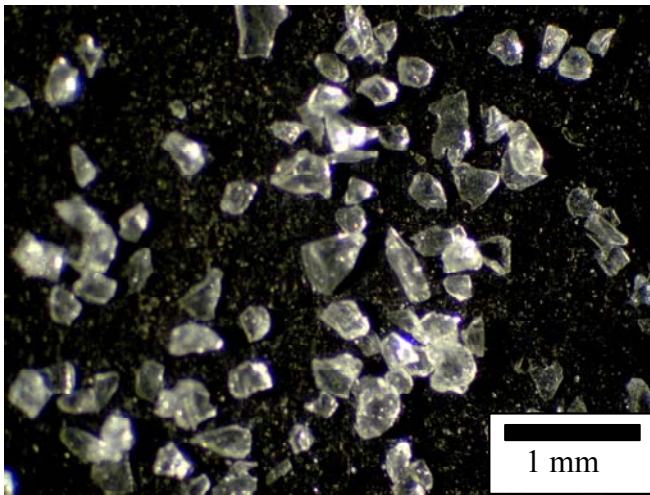
⁶ Timoshenko, S., Strength of Materials, Part II, Robert Krieger Publ. Co., Huntington, NY, 1976, p97.

ANNEX 2: GRANULOMETRY

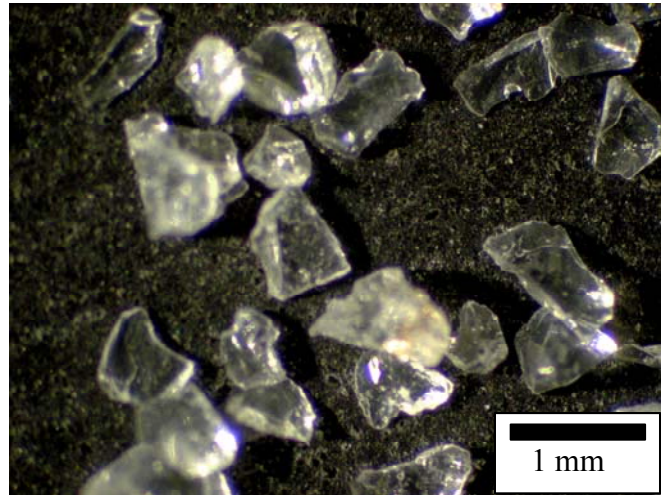
This work was undertaken by Minco in their US laboratories

A2.1 Images of coarser materials

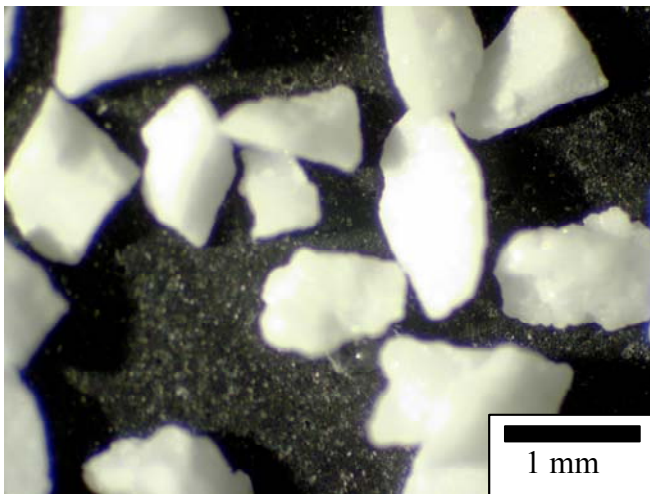
The following images have been prepared to clarify any issues of size distribution and shape.



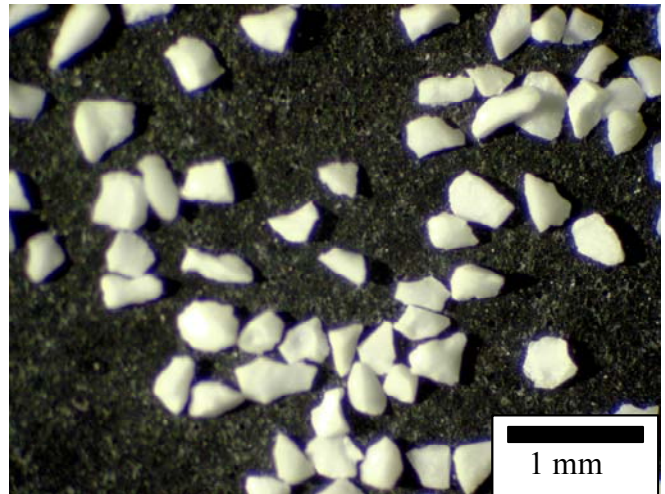
Studio 2, Min-Sil fused silica -50/+100 mesh



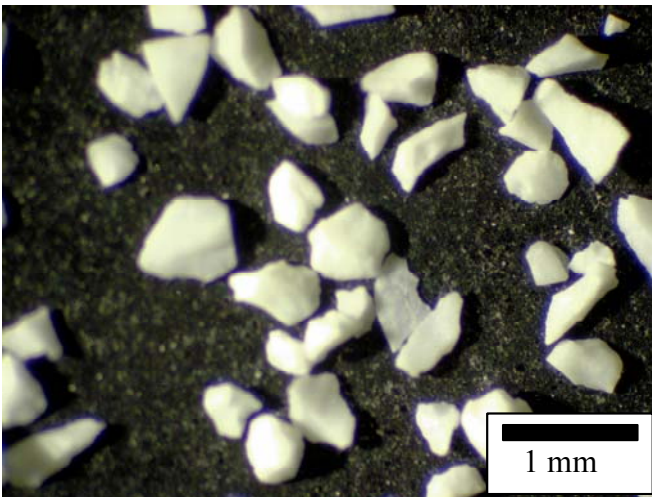
Studio 3, Min-Sil -30/+50 mesh



Studio 4, Tabular alumina 14/28 mesh



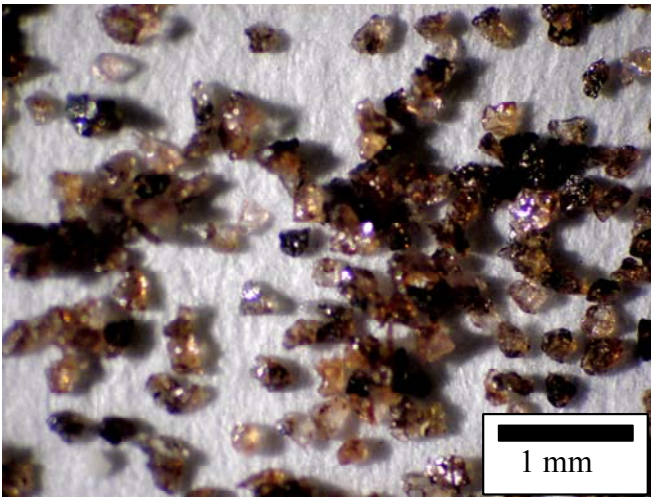
Studio 8, molochite 50/80 DD mesh



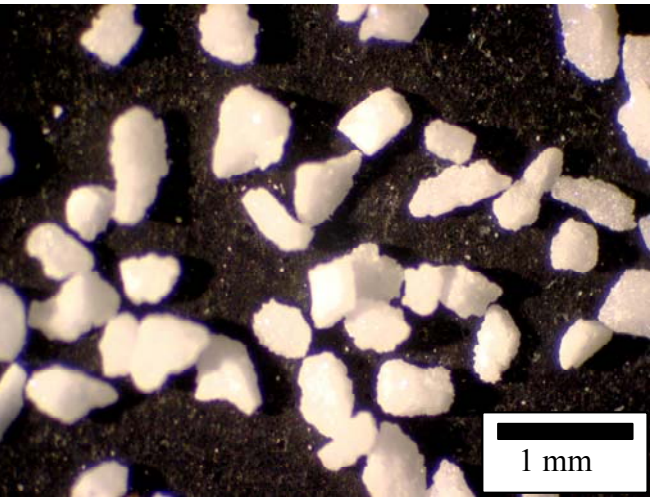
Studio 9, molochite, 30/80 DD mesh



Studio 10, molochite 16-30 DD mesh



Studio 11, AL80 brown fused alumina, 80 mesh



Studio 12, tabular alumina AL+60, 28-48 mesh



Studio 13, calcined zircon, 80 mesh

A2.2 Particle size distribution of coarse materials by sieving

Minco UK Ltd arranged for the measurement of the particle size distributions of the raw materials used by the University of Birmingham using dry sieving through a series of US screen sizes. The series was as follows: 20, 25, 30, 35, 40, 45, 50, 60, 70, 80, 100, 120, 140, 170, 200, 230, 270, 325, covering the range 0.0331" (850 μm) down to 0.0017" (45 μm) when converted to the mesh openings.

The sieve fractions obtained are given numerically in Table A2.2.1 and plotted in Figure A2.2.1. The cumulative size distributions are shown in Figure A2.2.2.

Table A2.2.1: Sieve fractions of coarser materials

US Sieve No.	Mesh opening, in	Mesh opening μm	Mass % retained								
			Studio 2, Mil-Sil -50/+100	Studio 3, Min-Sil -30/+50	Studio 4, Tabular alumina 14/28	Studio 8, Molochite 50/80	Studio 9, Molochite 30/80	Studio 10, Molochite 18/30	Studio 11, Brown fused alumina AL80	Studio 12, Tabular alumina AL60	Studio 13, Calcined Zircon 80
20	0.0331	841	0.00	0.21	46.38	0.00	0.00	27.73	0.00	0.00	0.00
25	0.0278	706	0.00	3.84	24.86	0.00	0.00	26.36	0.00	0.00	0.00
30	0.0234	594	0.00	19.53	17.51	0.00	0.00	19.72	0.00	0.00	0.00
35	0.0197	500	0.00	24.24	9.18	0.00	0.89	17.70	0.00	2.29	0.00
40	0.0165	419	0.00	17.58	1.50	0.06	11.64	5.66	0.00	13.33	0.00
45	0.0139	353	0.05	21.10	0.41	0.21	31.27	1.99	0.00	31.60	0.00
50	0.0117	297	2.34	9.45	0.08	0.37	23.49	0.49	0.01	33.70	0.01
60	0.0098	249	20.46	3.17	0.03	20.06	23.13	0.26	0.07	18.05	0.21
70	0.0083	211	25.74	0.59	0.03	53.10	7.73	0.06	12.94	0.92	1.76
80	0.007	178	21.69	0.18	0.01	20.26	1.51	0.00	60.64	0.07	10.30
100	0.0059	150	18.00	0.06	0.01	5.26	0.26	0.00	23.41	0.01	37.45
120	0.0049	124	8.88	0.00	0.00	0.58	0.00	0.00	2.81	0.00	26.80
140	0.0041	104	1.78	0.00	0.00	0.07	0.00	0.00	0.1	0.00	10.800
170	0.0035	88.9	0.64	0.00	0.00	0.02	0.00	0.00	0.01	0.00	7.48
200	0.0029	73.7	0.20	0.00	0.00	0.00	0.00	0.00	0.00	0.00	3.01
230	0.0025	63.5	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	1.13
270	0.0021	53.3	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.38
325	0.0017	43.2	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.24
Pan	-	<43.2	0.23	0.03	0.02	0.00	0.08	0.01	0.00	0.04	0.43

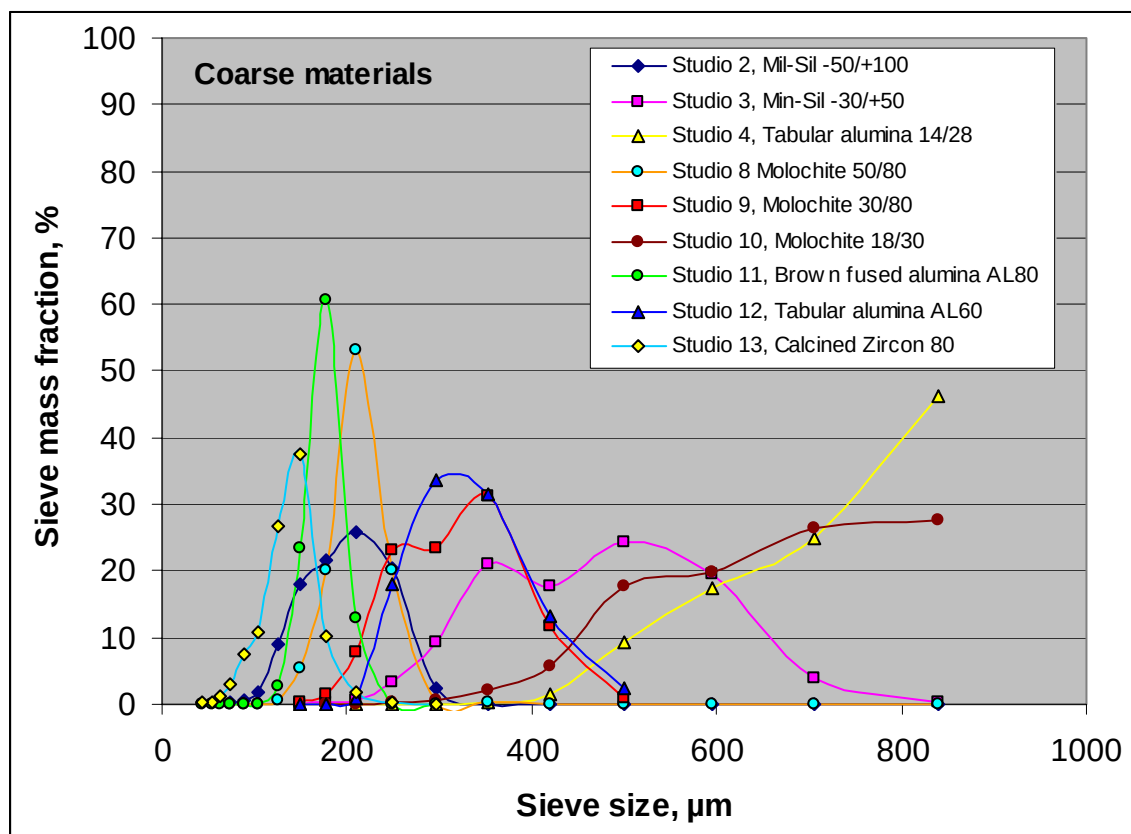


Figure A2.2.1: Coarser materials, size fractions by mass.

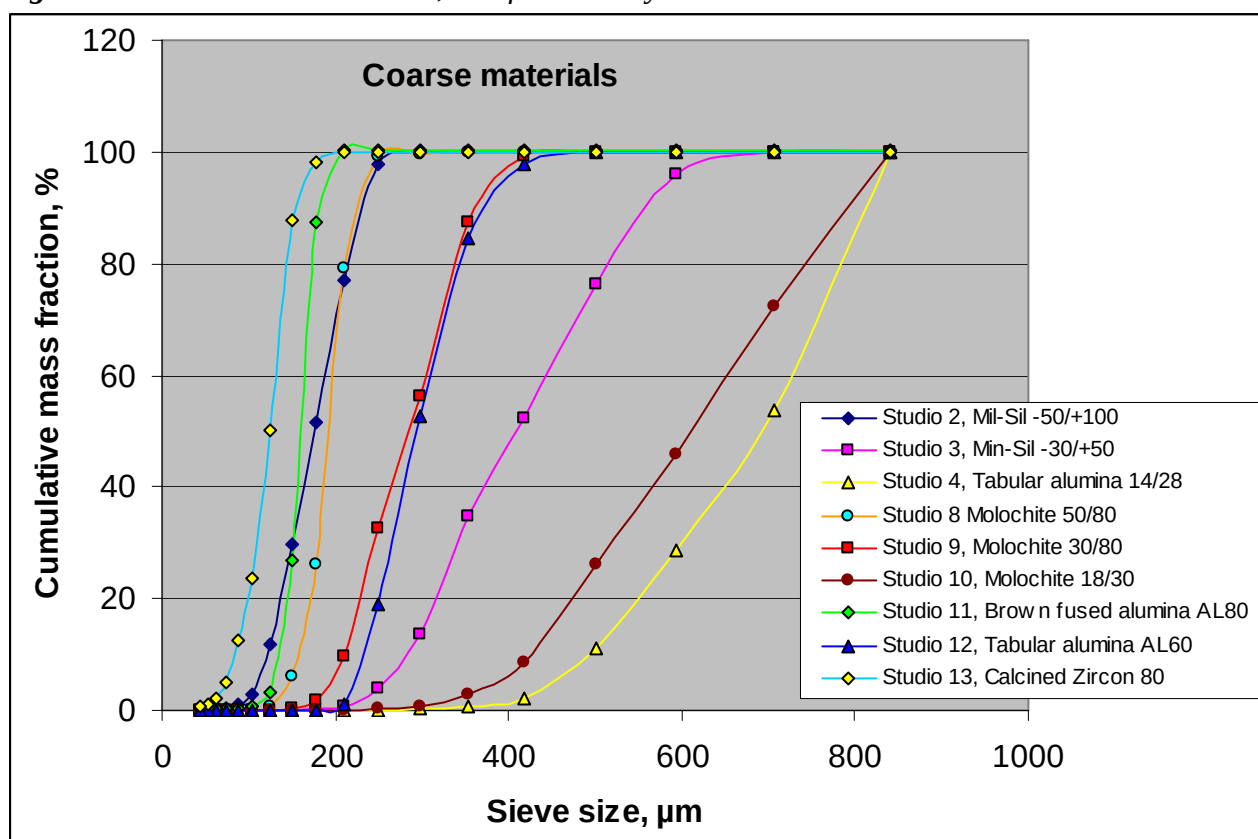


Figure A2.2.2: Coarser materials, cumulative size distributions by mass.

A2.3 Particle size distributions of finer materials

Minco UK Ltd also undertook to provide particle size information on the finer materials using both sieving and laser sizing using a Microtrac instrument. This was applied to four materials, Studio 1, 5, 6, and 7. The original data have been summarised in Tables A2.3.1 and A2.3.2, and are plotted in Figures A2.3.1 to A2.3.3.

Table A2.3.1: Sieve fractions of finer materials

(Note: Wet sieved through 325 mesh, trapped fraction dried and dry sieved through sieve series; high proportions are left in the pan passing the 325 mesh sieve.)

US sieve No.	Mesh opening, in	Mesh opening μm	Mass % retained			
			Studio 1, Min-Sil 120F	Studio 5, Fused white alumina 200	Studio 6, Zircosil 200	Studio 7, Molochite 200
50	0.0117	297	0.32	0.00	0.00	0.00
60	0.0098	249	0.36	0.00	0.00	0.00
70	0.0083	211	0.60	0.00	0.00	0.00
80	0.007	178	1.04	0.00	0.02	0.22
100	0.0059	150	2.10	0.08	0.02	0.20
120	0.0049	124	5.02	0.47	0.06	0.36
140	0.0041	104	5.04	4.20	0.02	0.52
170	0.0035	88.9	8.28	6.88	0.06	1.10
200	0.0029	73.7	7.68	6.94	0.22	1.98
230	0.0025	63.5	8.96	7.88	1.08	2.46
270	0.0021	53.3	6.36	6.46	2.34	2.86
325	0.0017	43.2	5.76	5.98	3.94	4.26
Pan		<43.18	48.48	61.11	92.24	84.14

Table A2.3.2: Microtrac analyses

Microtrac channel, μm	Cumulative volume % passing				Volume % in size increment			
	Studio 1, Min-Sil 120F	Studio 5, Fused white alumina 200	Studio 6, Zircosil 200M	Studio 7, Molochite 200	Studio 1, Min-Sil 120F	Studio 5, Fused white alumina 200	Studio 6, Zircosil 200M	Studio 7, Molochite 200
704	100	100	100	100	0.03	0.00	0.00	0.00
497.8	99.97	100	100	100	0.51	0.00	0.00	0.00
352	99.46	100	100	100	1.10	0.01	0.00	0.01
248.9	98.36	99.99	100	99.99	4.93	1.26	0.00	0.30
176	93.43	98.73	100	99.69	9.98	6.82	0.00	1.02
124.5	83.45	91.91	100	98.67	13.09	11.75	0.16	3.06
88	70.36	80.16	99.84	95.61	13.43	13.05	3.75	7.03
62.23	56.93	67.11	96.09	88.58	12.26	12.7	9.30	10.87
44	44.67	54.41	86.79	77.71	10.38	12.00	12.81	13.28
31.11	34.29	42.41	73.98	64.43	7.70	9.64	11.53	11.94
22	26.59	32.77	62.45	52.49	6.37	8.26	10.40	11.05
15.56	20.22	24.51	52.05	41.44	5.22	6.76	9.35	9.56
11	15.00	17.75	42.7	31.88	3.63	4.37	7.23	6.86
7.778	11.37	13.38	35.47	25.02	3.13	3.50	6.35	5.96
5.5	8.24	9.88	29.12	19.06	3.08	2.96	5.74	5.30
3.889	5.16	6.92	23.38	13.76	2.44	1.74	2.98	3.97
2.75	2.72	5.18	20.4	9.79	1.81	2.15	5.63	4.26
1.945	0.91	3.03	14.77	5.53	0.67	1.64	6.43	2.87
1.375	0.24	1.39	8.34	2.66	0.24	1.15	5.61	2.04
0.972	0.00	0.24	2.73	0.62	0.00	0.24	2.73	0.62

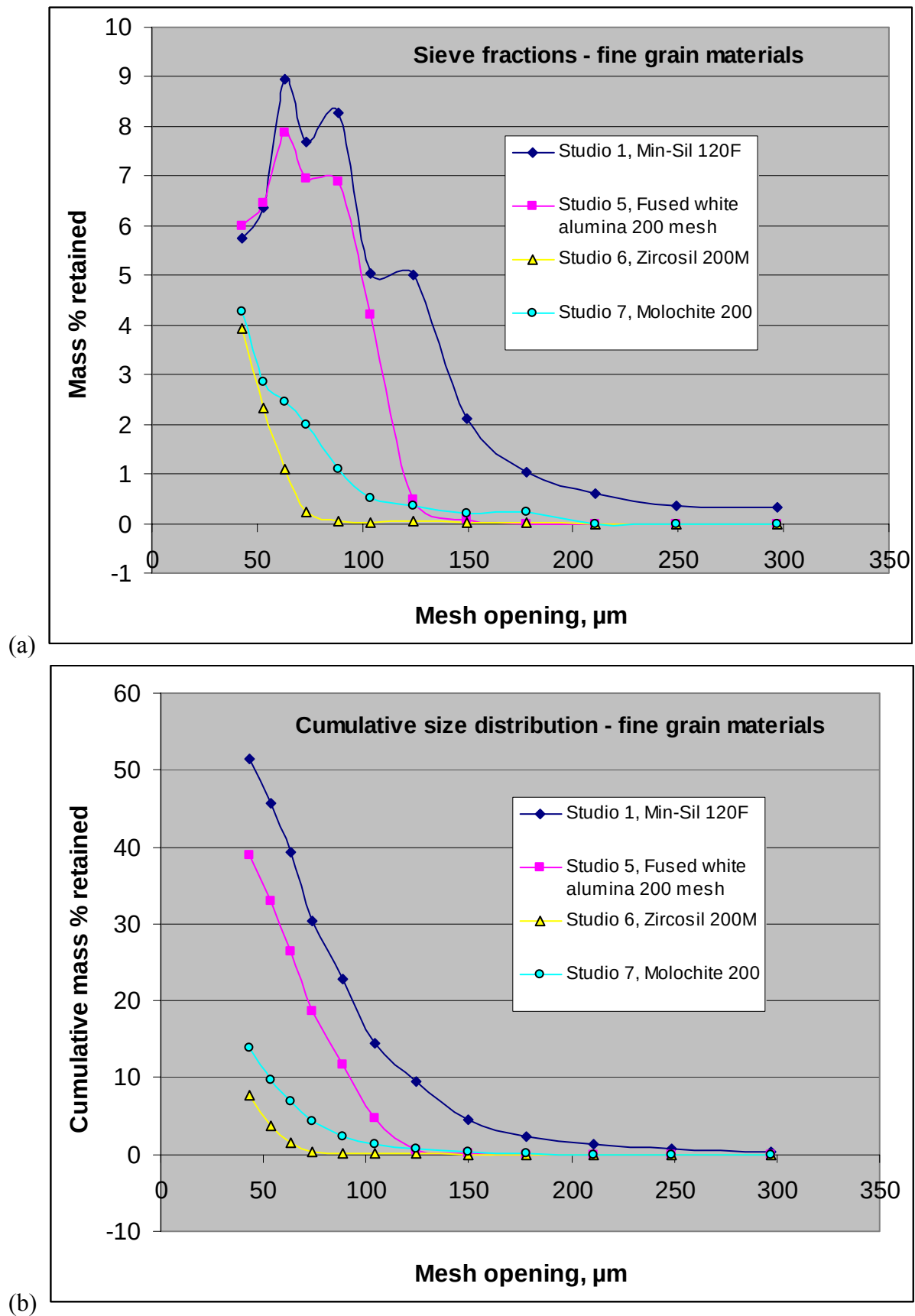


Figure A2.3.1: Fine materials, sieve fractions, (a) sieve fraction, mass %, (b) cumulative distribution, mass %

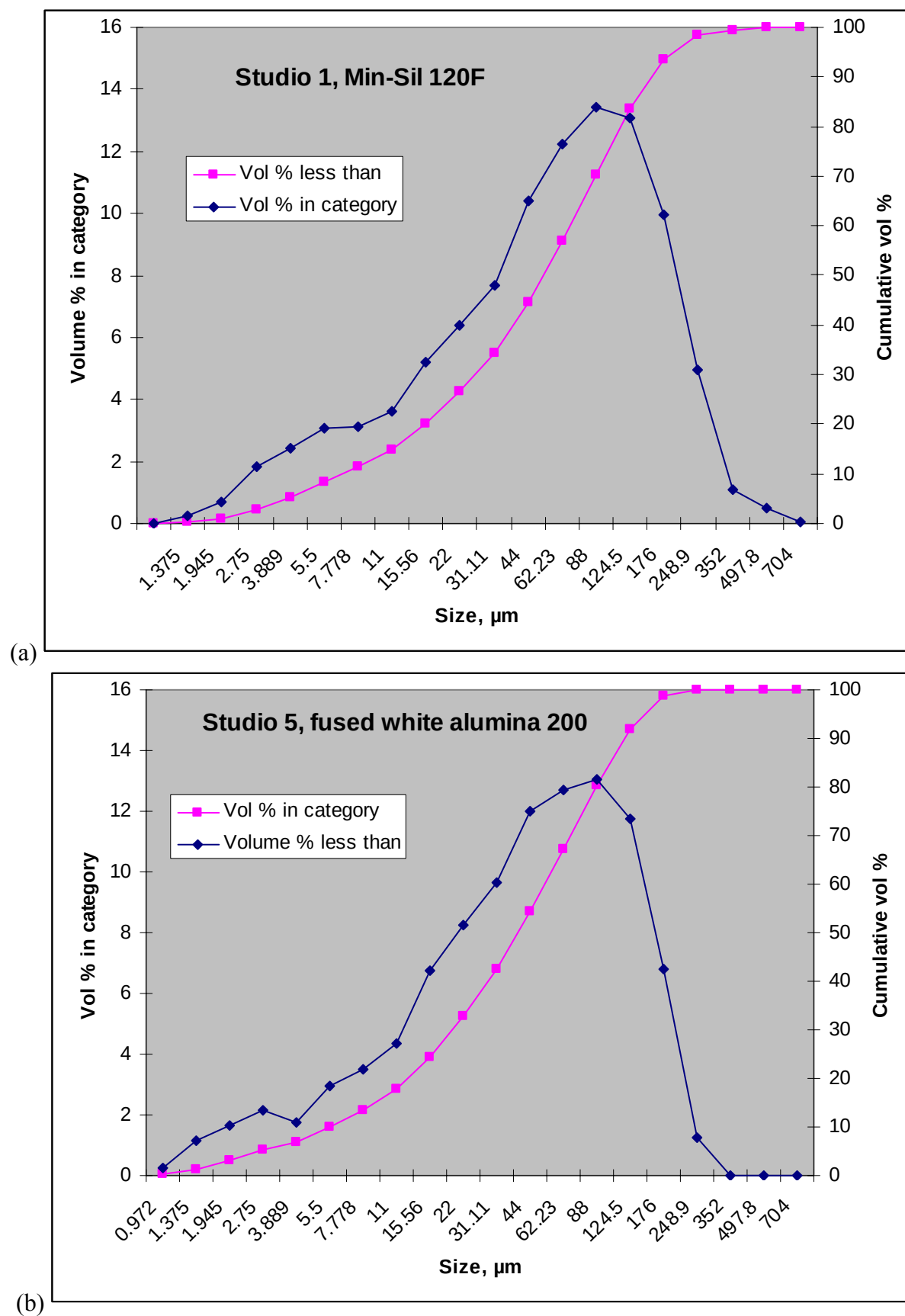


Figure A2.3.3: Microtrac particle size distributions for (a) Studio 1, (b) Studio 5 powders.

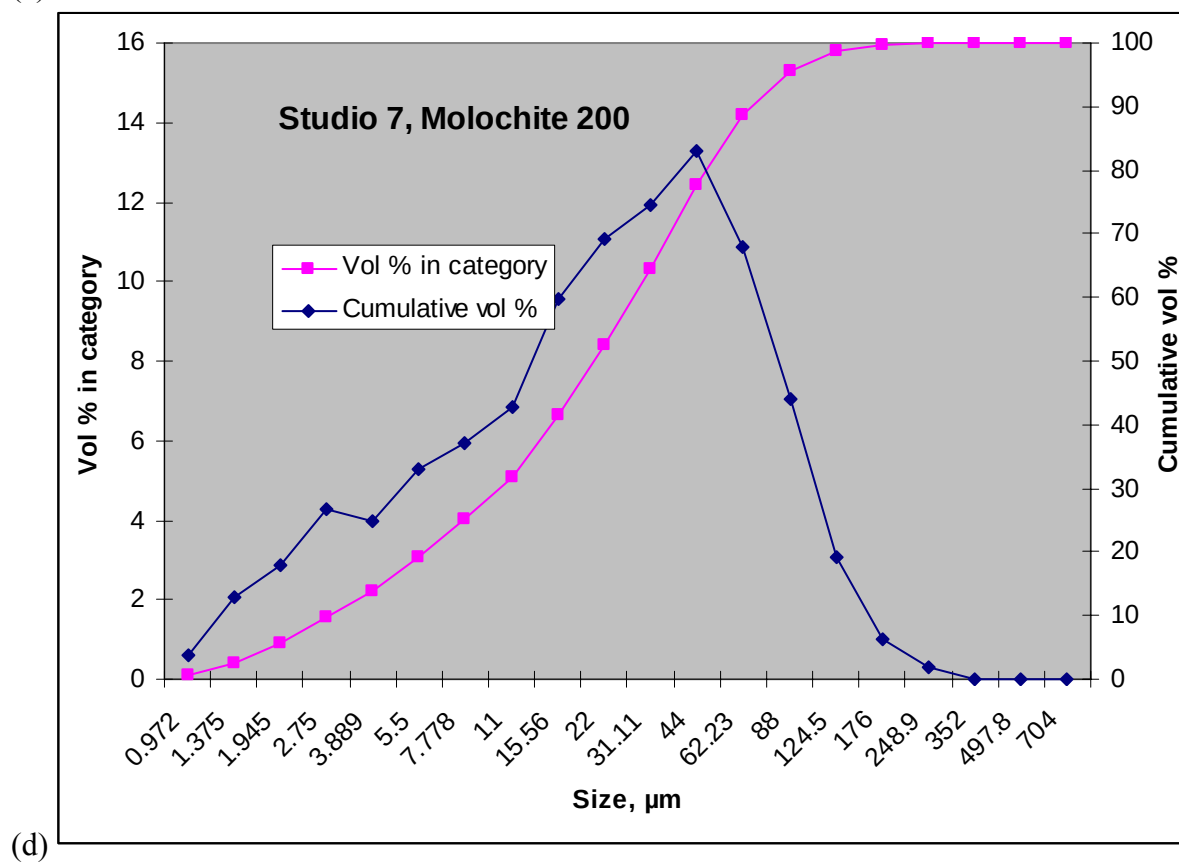
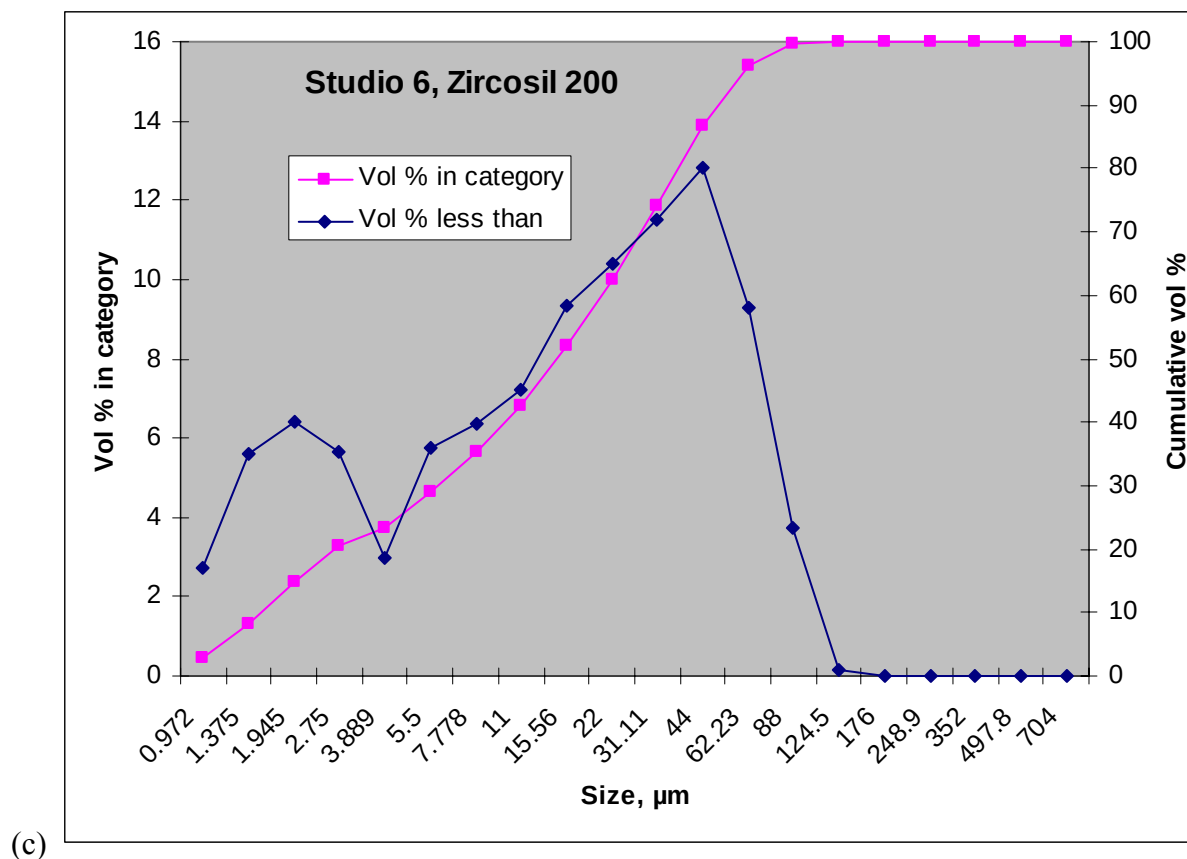


Figure A2.3.3 (cont.): Microtrac particle size distributions for (c) Studio 6, (d) Studio 7.

A2.4 Specific surface area measurements

Specific surface area measurements (Table A2.4.1) were made by the BET method, supplied through Minco UK Ltd.

Table A2.4.1 Specific surface area data for finer materials with particle sizing equivalents

Material	Specific surface area, m²/g	Density, Mg m⁻³	Equivalent spherical diam., µm	Number average size, Microtrac, µm
Studio 1, Mil-Sil 120F	0.46	2.5	5.22	6.64
Studio 5, Fused white alumina 200	0.65	3.98	2.32	4.51
Studio 6, Zircosil 200	1.66	4.5	0.80	2.50
Studio 7, Molochite 200	1.42	2.9	1.46	3.58

In the above table, the density has been assumed as that of solid material, and has been used to compute a mean equivalent spherical particle diameter from the specific surface area. In addition, from the Microtrac particle size distributions, the mean particle size has been determined on a number basis from the 'channel size' (Table A2.3.2). It can be seen that while there is a reasonable correlation between these two methods of sizing, there is a significant difference between the respective values which increases with declining particle size. Note that:

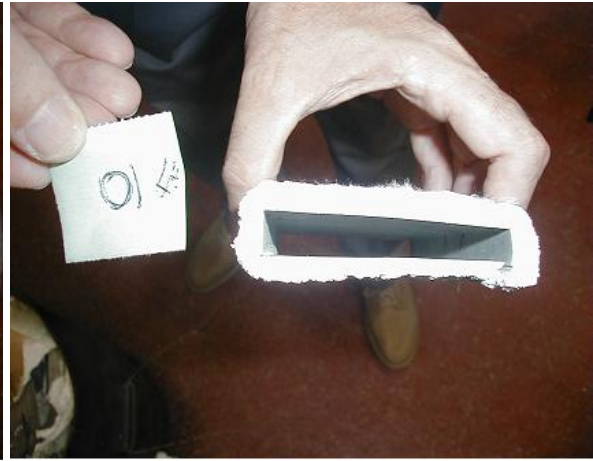
1. The 'channel size' will overestimate the 'equivalent spherical diameter' leading to an overestimate of the average size;
2. It is believed that only the Fraunhofer light-scattering theory has been used for the Microtrac analysis, and that
3. Particle shape factors have been ignored.
4. For Studio 6 material, there was a significant volume fraction of material in the smallest size category.

ANNEX 3: PLATE BULGE TEST DIMENSIONAL MEASUREMENTS

Not all casting resulted in satisfactory plates (Figure A3.1).



System 1, Full silica 1, NPL01, runner



System 1, Full silica, NPL01, single mould



System 2, Full aluminosilicate 1, NPL03



System 2, Full aluminosilicate 1, NPL04



System 3, Zircon/molochite 1, NPL07



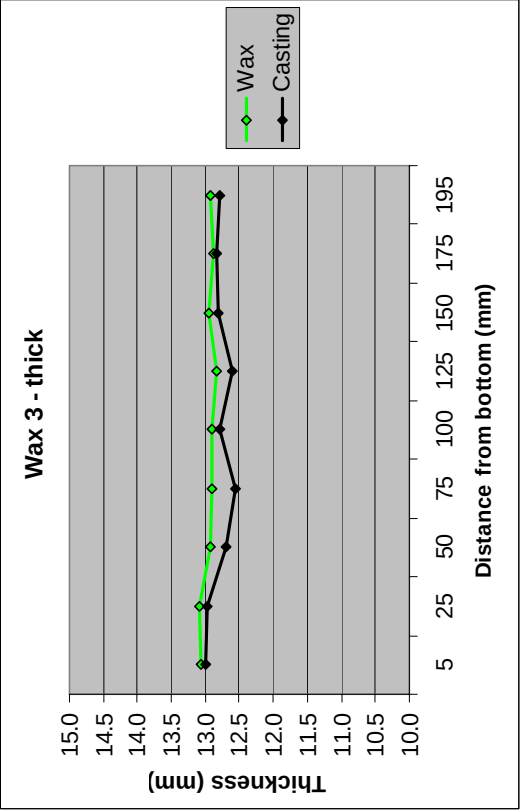
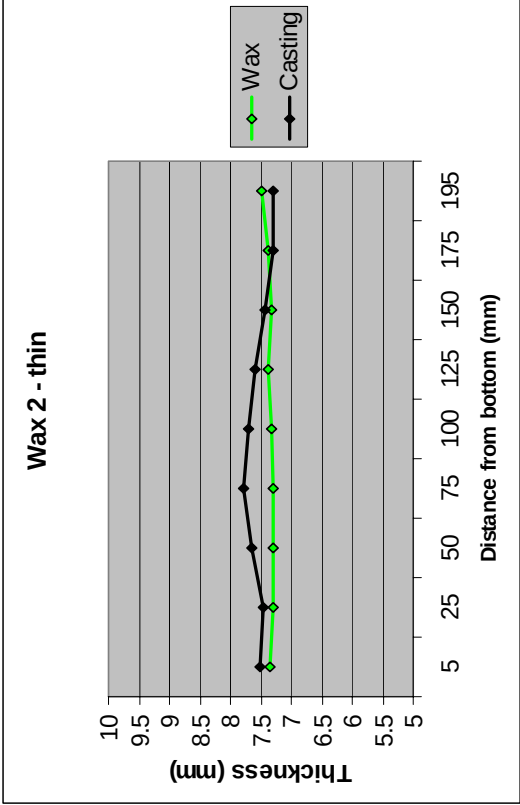
System 4, Zircon/alumina 1, NPL17

Figure A3.1: Thinner mould systems after casting, showing transient bulging before failure.

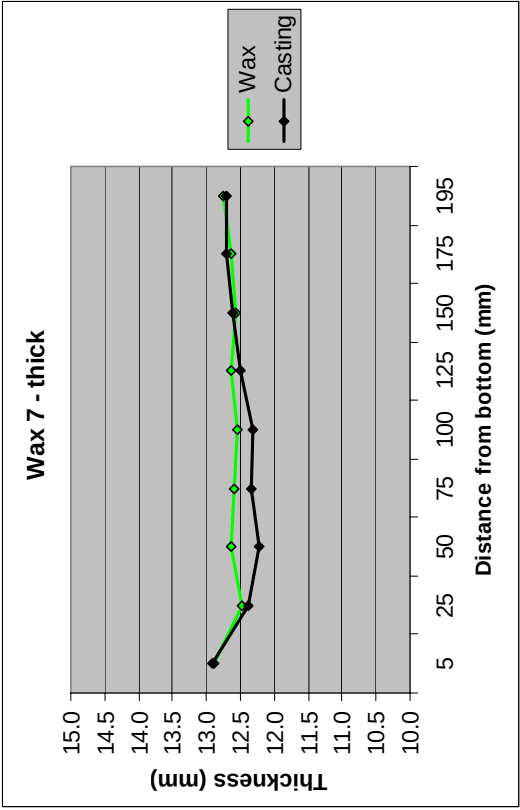
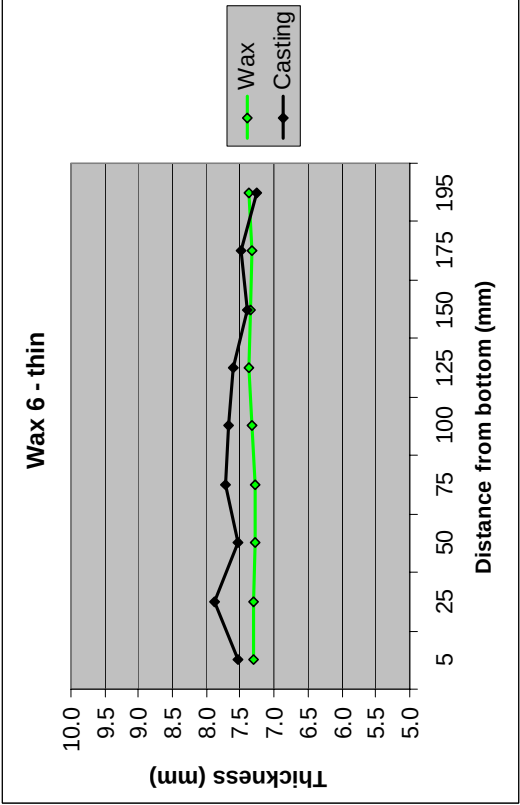
The following plots show the plate thickness determined along the single line shown in Figure 37 as measured on the wax and on the subsequent cast metal shape.

Full silica system 1:

NPL01 – large runner

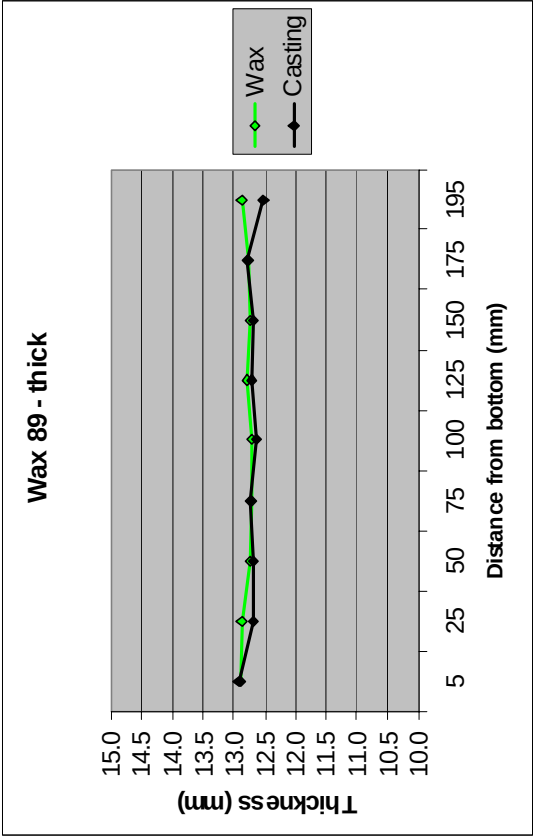
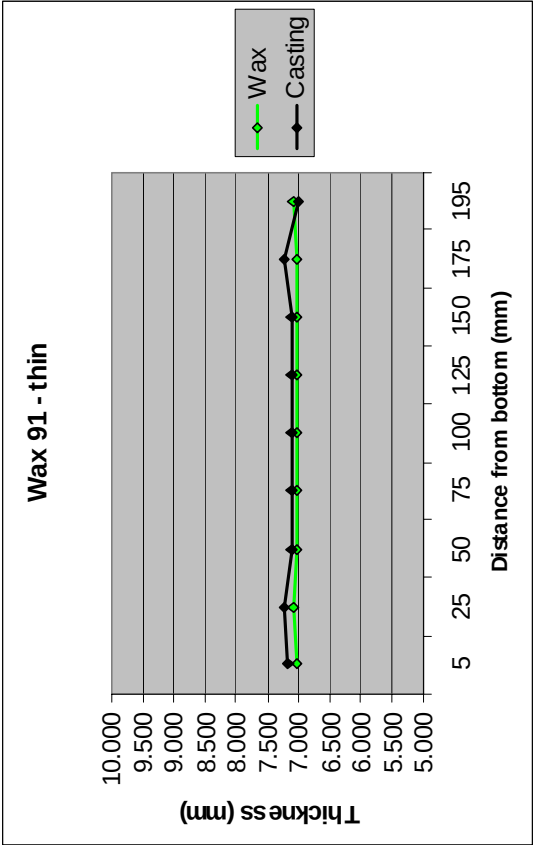


NPL02 – small runner:

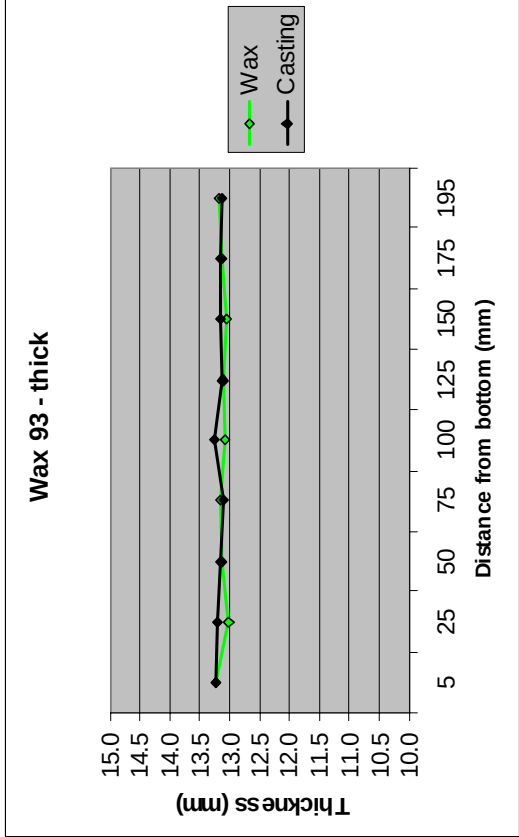
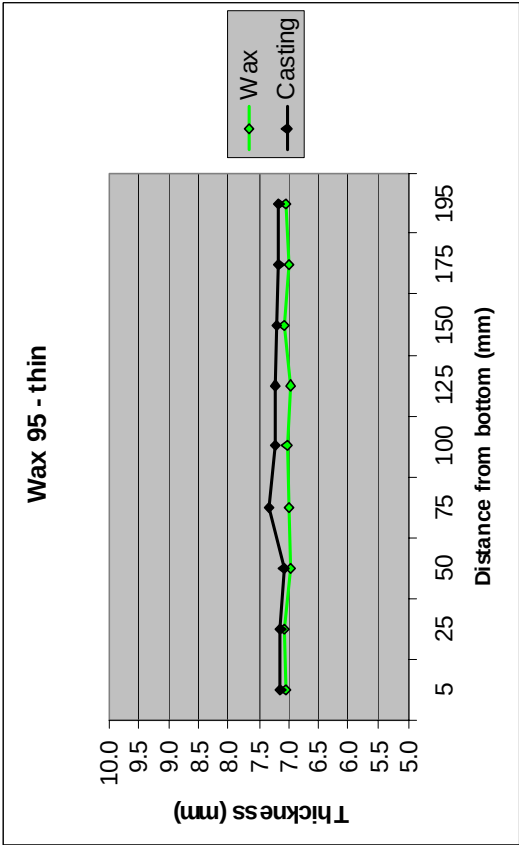


Full silica system 2:

NPL23 – large runner

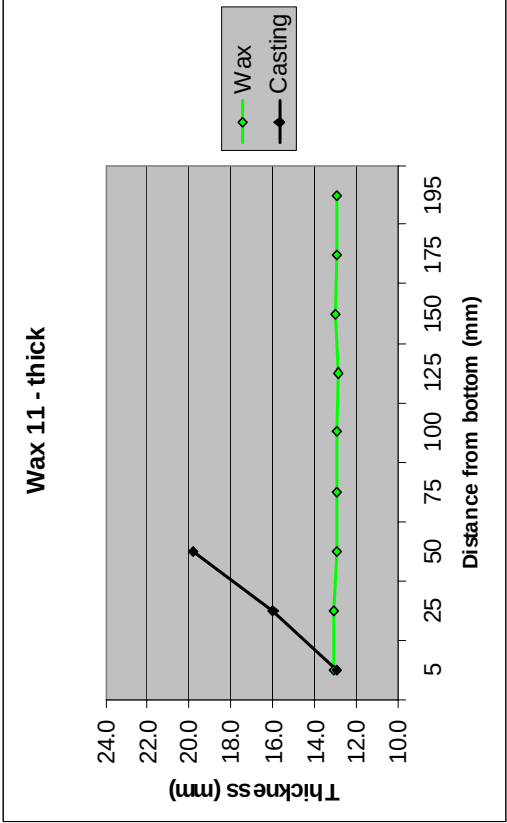
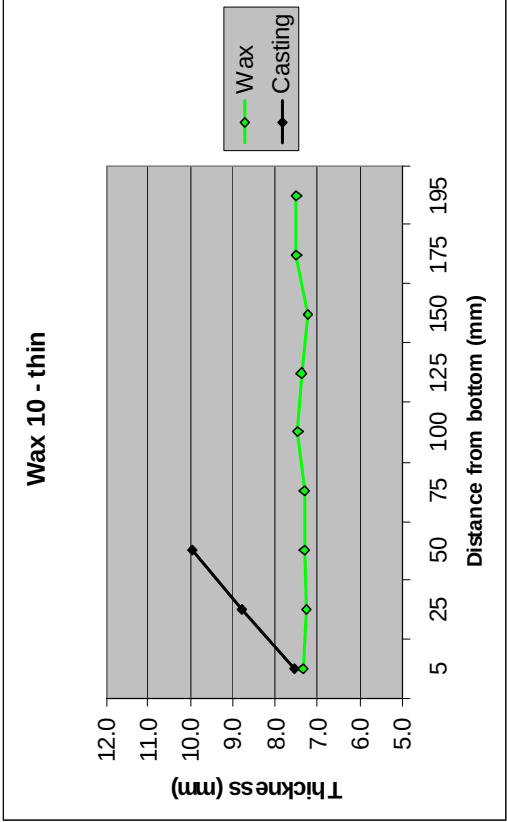


NPL24 – small runner

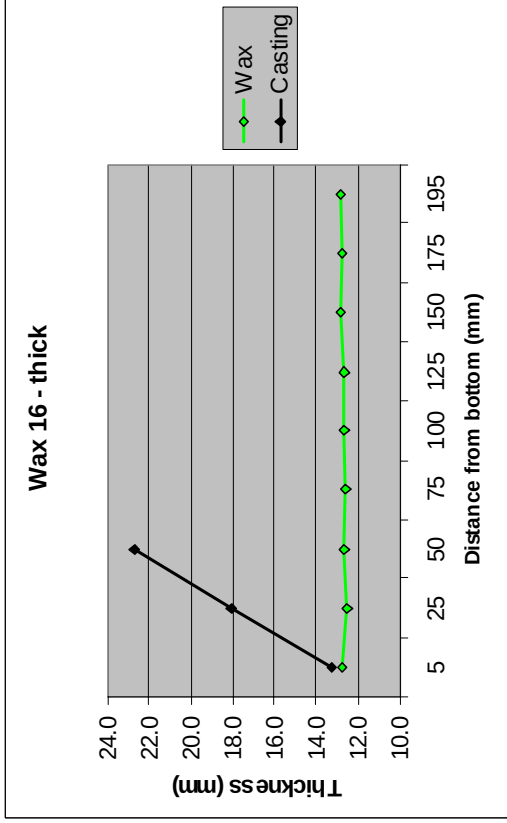
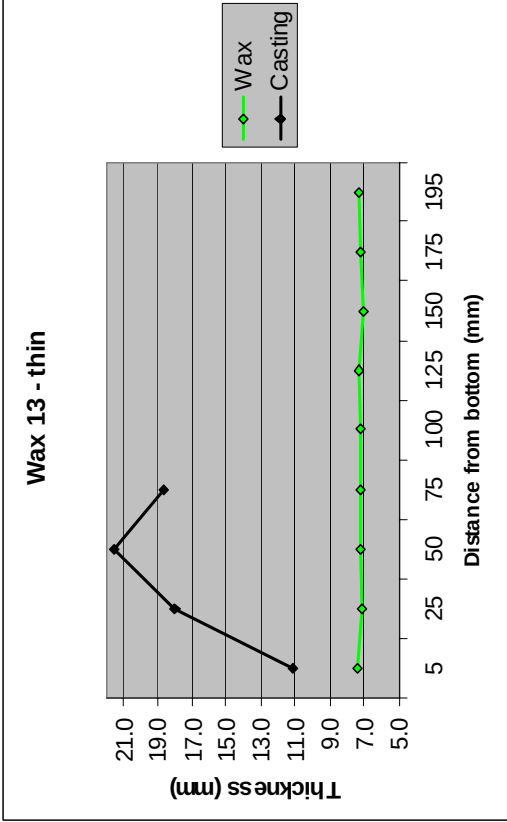


Full aluminosilicate system 1

NPL03, large runner

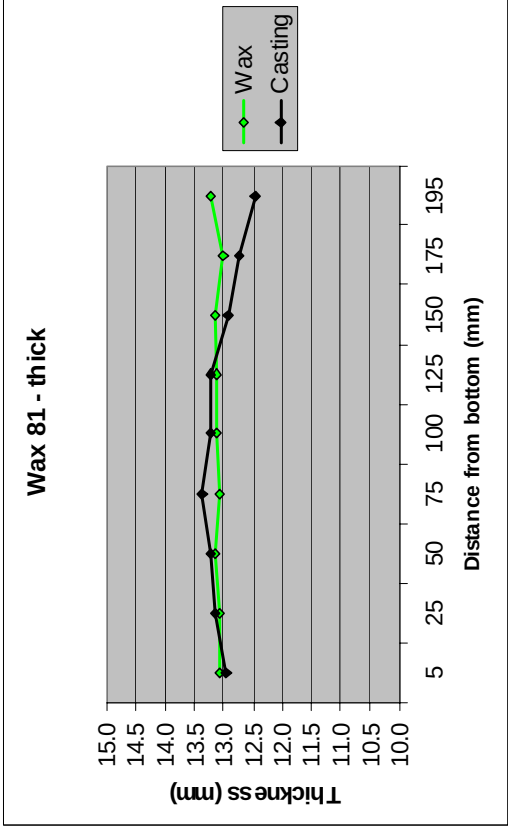
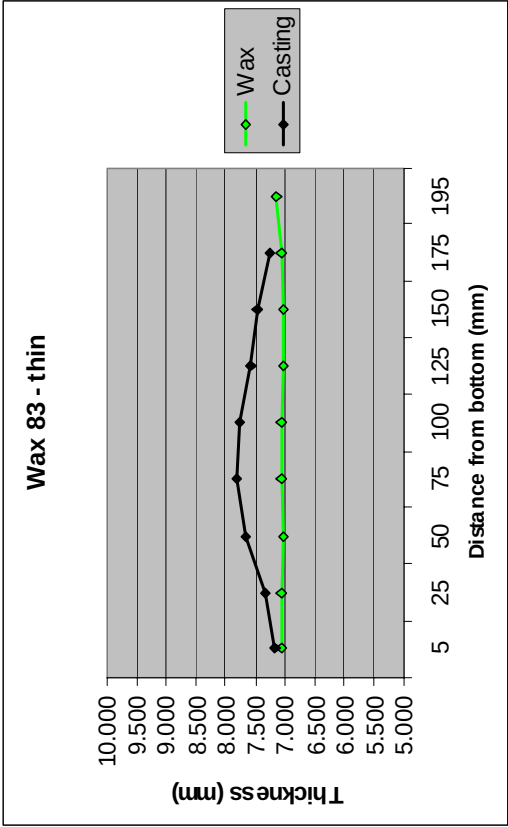


NPL04, small runner

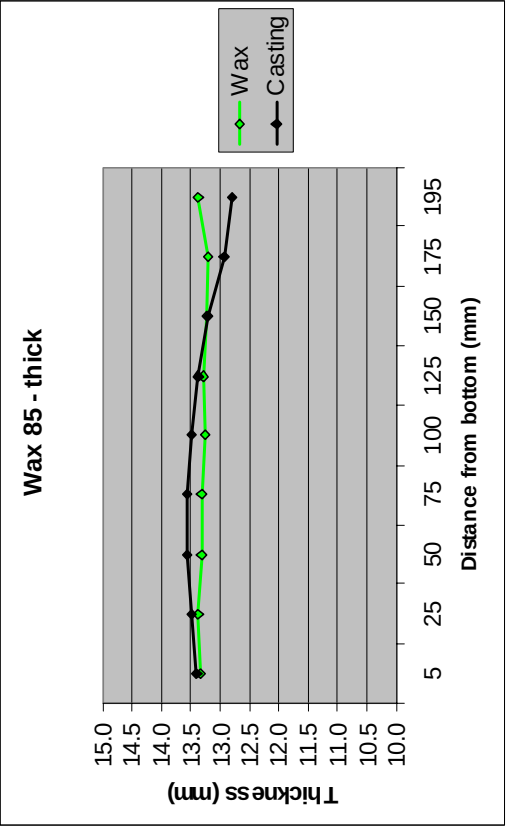
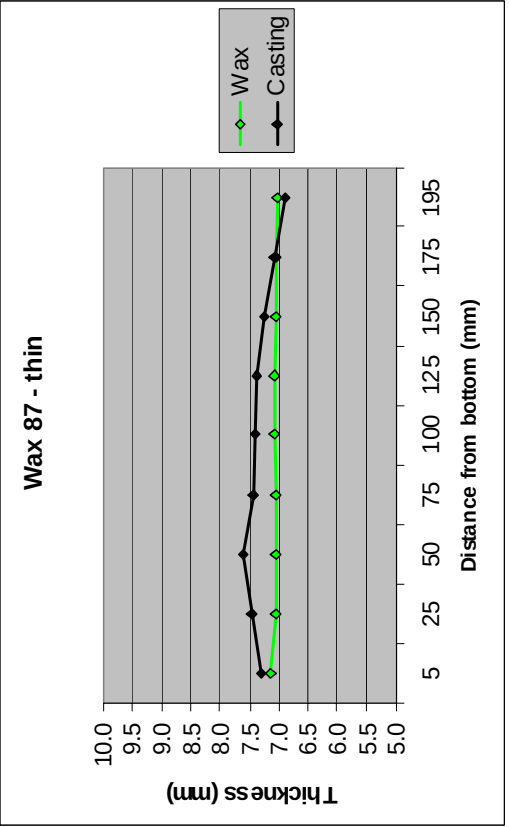


Full aluminosilicate 2

NPL21, large runner

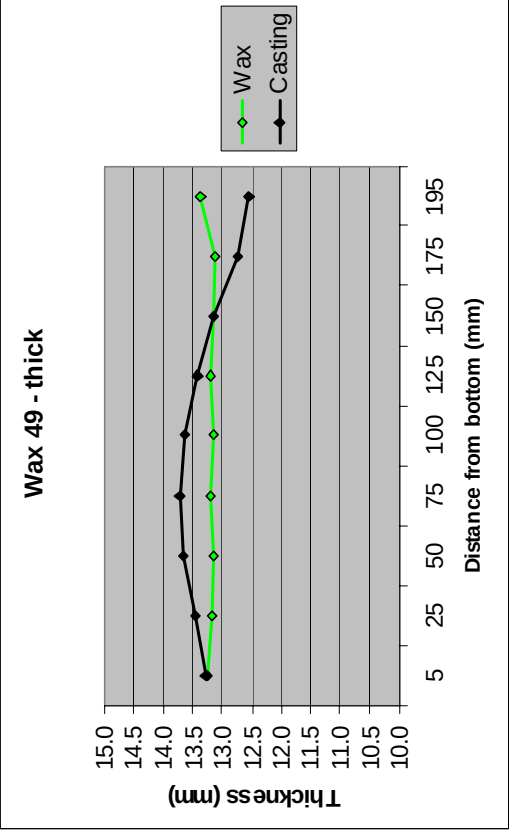
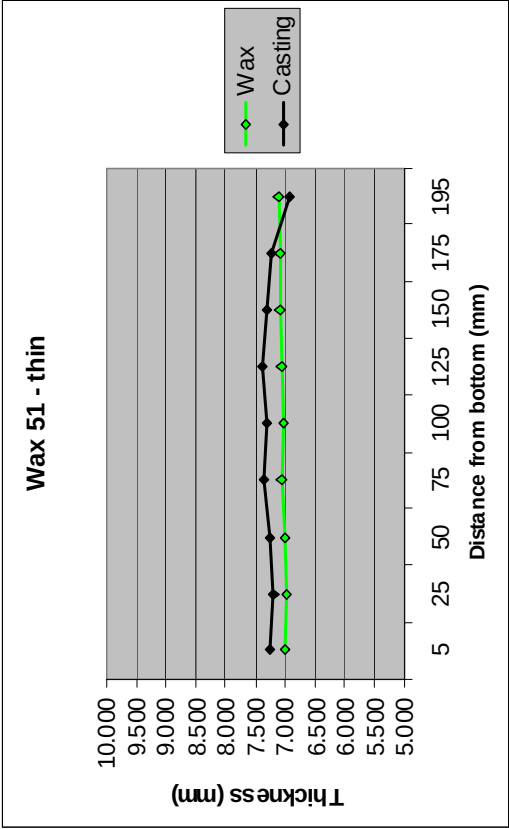


NPL22, small runner

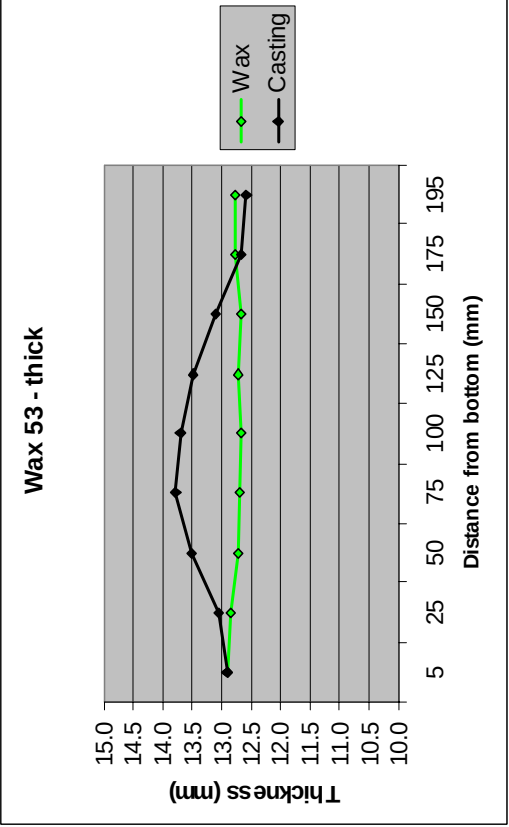
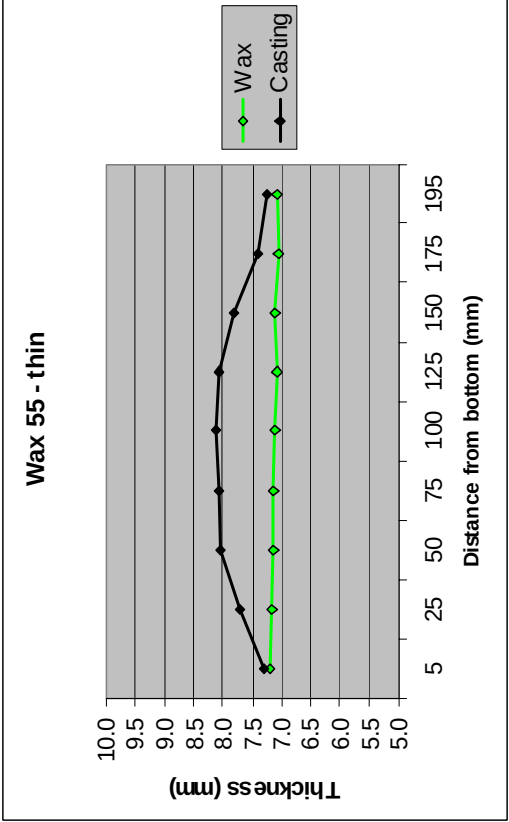


Zircon/molochite system 2 (note: no results from zircon/molochite system 1)

NPL13, large runner system

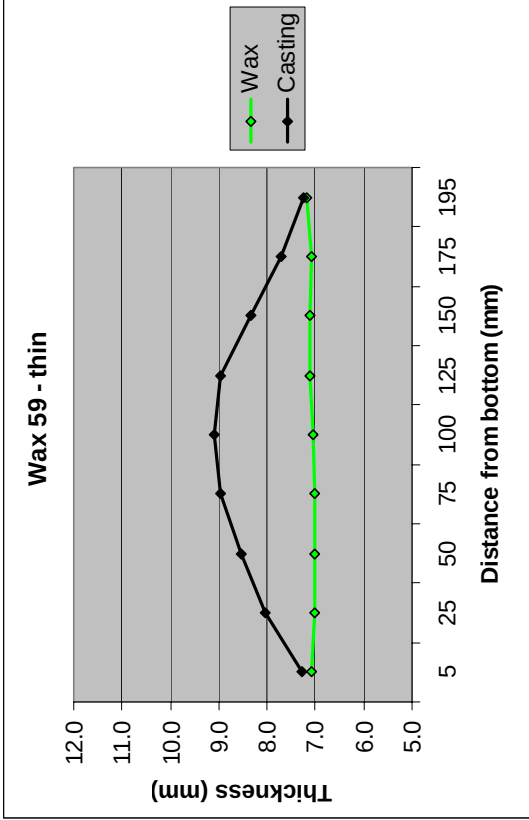


NPL14, small runner system



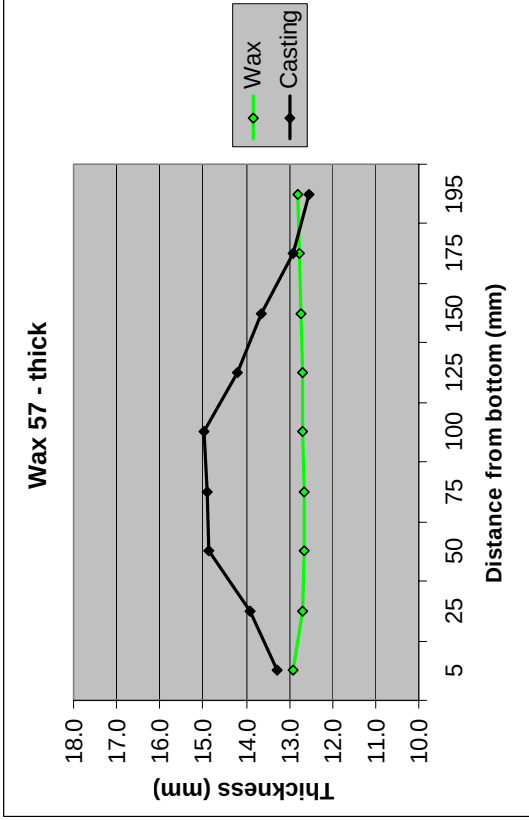
Zircon/alumina system 2 (note no results from zircon/alumina system 1)

NPL15, large runner



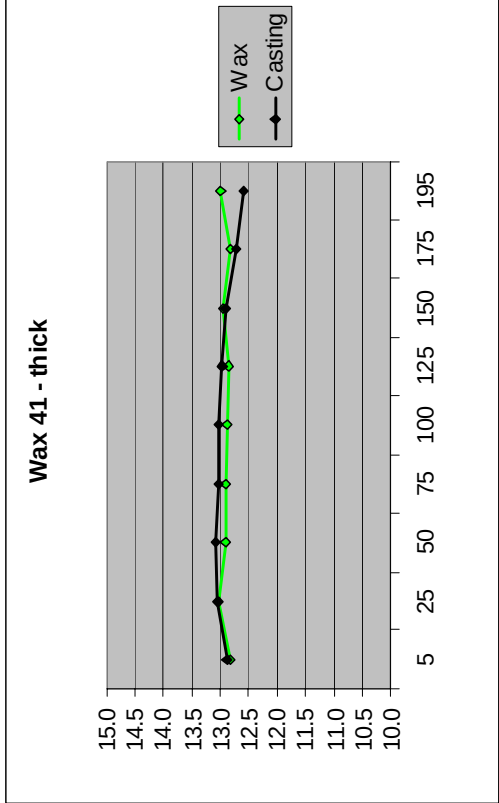
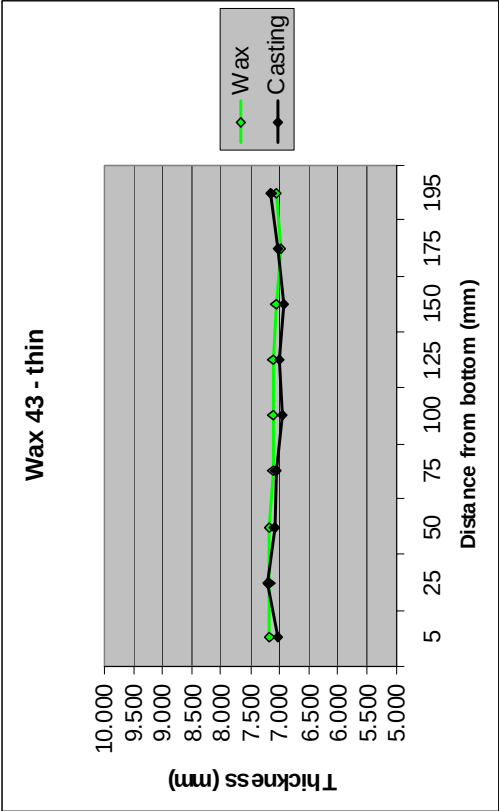
NPL16, small runner,

No results

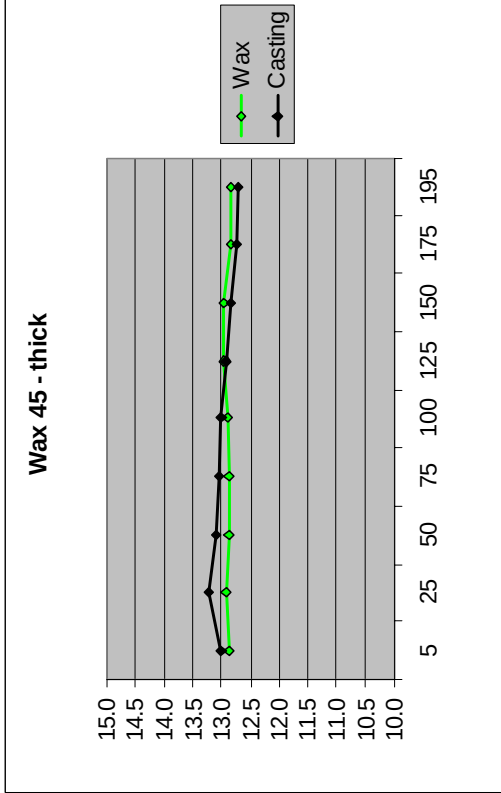
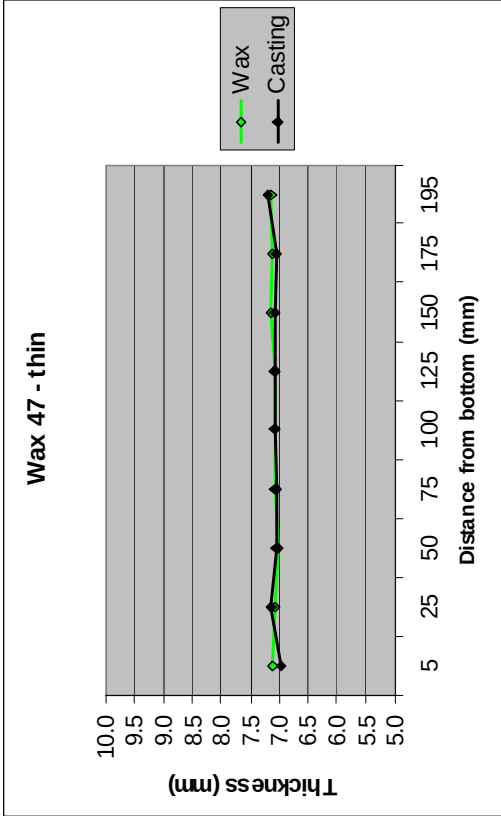


Partner 1 mould

NPL11, large runner

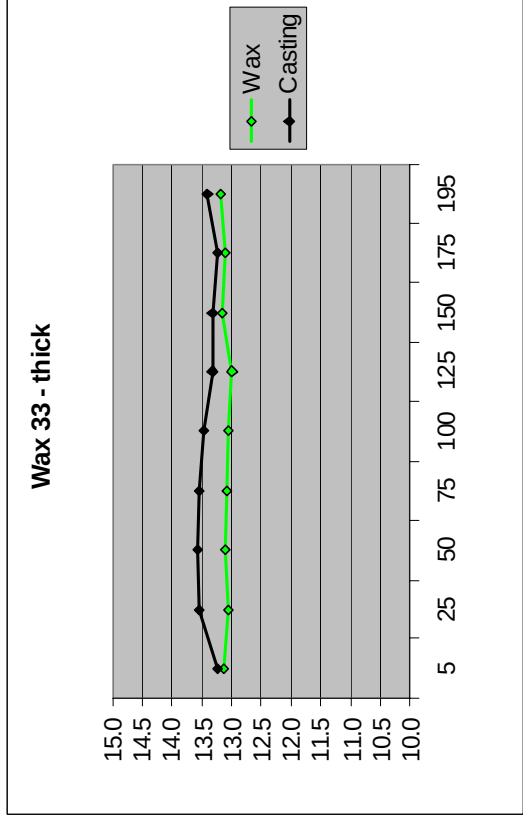
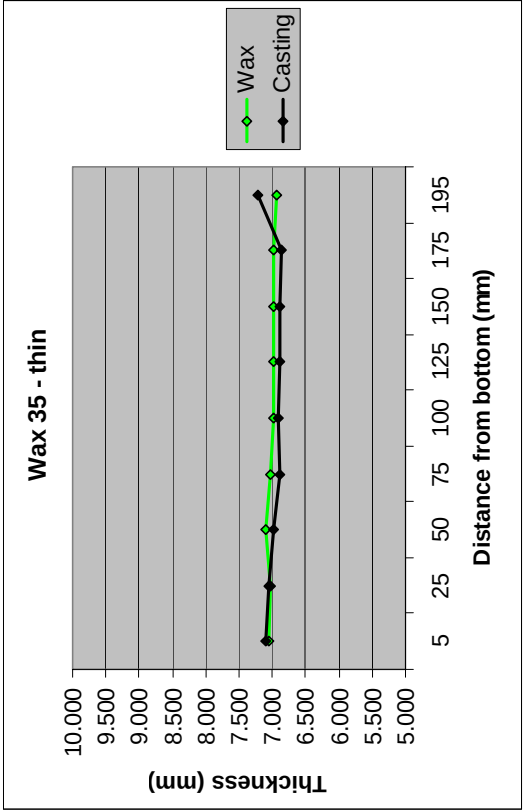


NPL12, small runner

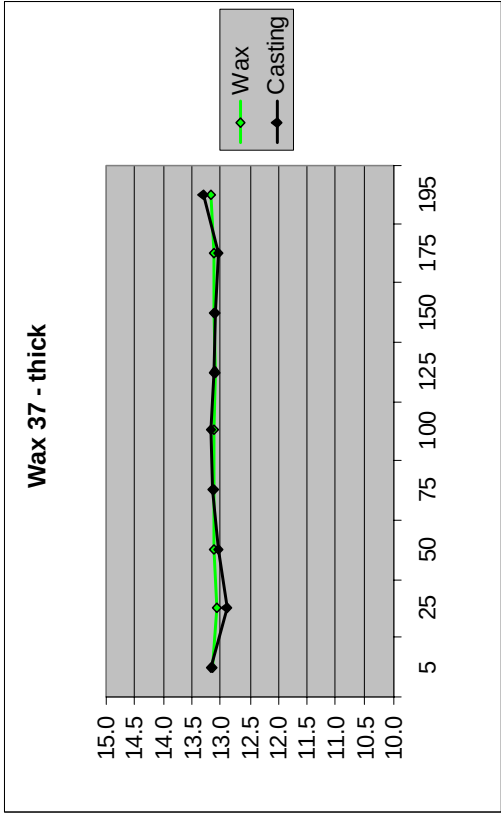
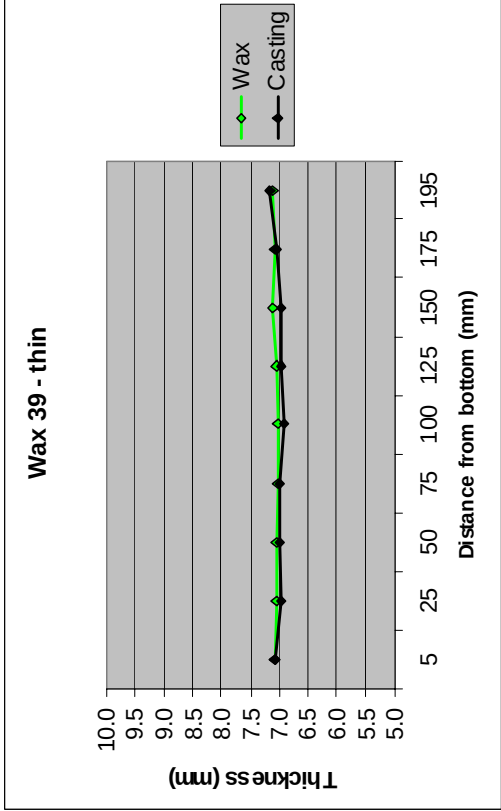


Partner 2 mould

NPL09, large runner

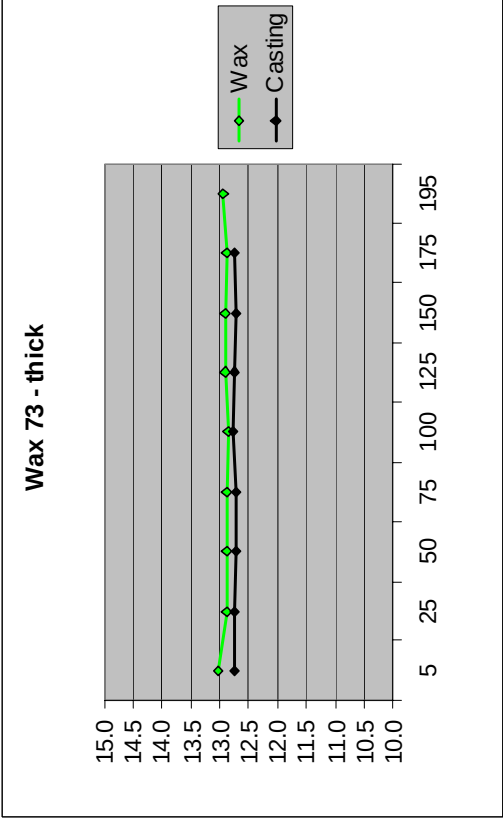
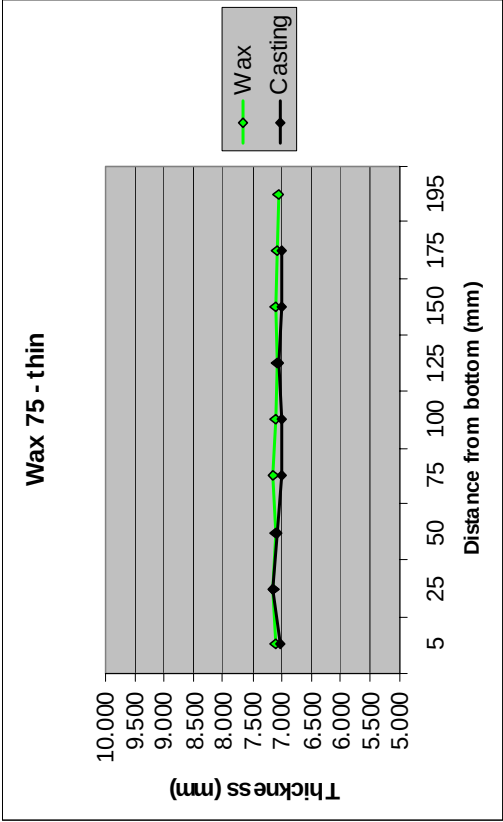


NPL10, small runner



Partner 3 mould

NPL19, larger runner



NPL20, small runner

No results

