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(54) Title: A METHOD FOR PRODUCING A MASONRY UNIT AND A MASONRY STRUCTURE FORMED FROM SAID MASONRY UNIT

(57) Abstract: The present invention relates to a method for producing a construction element comprising forming a mixture of a bituminous binder with a course aggregate, compacting said mixture at a compaction level of up to around 7 MPa, and at least partially curing the compacted mixture. There is further provided a method for producing a masonry unit, the method comprising forming a mixture containing a graded aggregate and around 6 wt % of a bituminous binder, compacting the mixture, and at least partially curing the compacted mixture. Another aspect of the present invention provides a method for producing a masonry unit, the method comprising forming a mixture containing a graded aggregate and a bituminous binder, compacting the mixture, and at least partially curing the compacted mixture by heating the compacted mixture to a temperature of around 200 °C. The present invention further relates to a masonry structure comprising a plurality of masonry units and a mortar, at least one of said masonry units comprising a graded aggregate and a bituminous binder.

# A METHOD FOR PRODUCING A MASONRY UNIT AND A MASONRY STRUCTURE FORMED FROM SAID MASONRY UNIT

The present invention relates to methods for producing masonry units, particularly, but not exclusively, environmentally friendly masonry units, and masonry structures formed from said masonry units.

The developed world has a major problem – it is running out of space to store all of the rubbish it creates. The current trend for land-filling is simply unsustainable. By way of example, in March 2005 it was estimated that the UK had 6 years disposal capacity remaining at current rates of tipping (Environment Agency 2005). In Europe, the situation is similar with high prices and taxes levied on material sent to landfill. The EU Landfill Directive has set stringent and demanding targets on all EU countries for reducing material sent to landfill (The Waste Thematic Strategy 2005).

A significant need therefore exists for a novel construction/masonry unit which could significantly contribute to the increased need for recycling; reduction in demand for natural aggregate extraction; reduction in waste sent to landfill; and/or the UK's 2016 zero-carbon new homes target (Communities and Local Government 2006). An object of the present invention is to address the above need.

According to a first aspect of the present invention there is provided a method for producing a masonry unit, the method comprising forming a mixture containing a bituminous binder and a graded aggregate containing a coarse aggregate fraction, compacting the mixture at a compaction level of up to around 7 MPa, and at least partially curing the compacted mixture.

The present invention provides a method for producing a masonry unit, hereinafter referred to generically as "Bitublock", which can be composed entirely of recycled/waste aggregates and binding agents from selected waste bitumens. As such, it could significantly contribute to a reduction in material sent to landfill. The performance of Bitublock has been shown to be at least equivalent to concrete block masonry products found in the UK. The present application describes the long-term behaviour of Bitublock masonry. These results have also been used to confirm the successful encapsulation of the recycled material by the bitumen binder. The masonry

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did not exhibit any enlarged expansion (cryptoflorescence) which results from chemical interaction between the mortar and Bitublock.

Bitublock readily satisfies all the physical and strength requirements of current coarse aggregate concrete block units manufactured in the UK but does not necessarily need to incorporate any natural aggregates and does not require any specifically manufactured or synthetic binders. Instead the new unit, Bitublock, can, if desired, incorporate only recycled aggregates. Bitublock is a masonry unit which may be composed entirely of recycled and waste aggregates and binding agents from selected commercially available and waste bitumens. Previously, the physical properties of Bitublock units composed of mixtures of incinerated sewage sludge ash, incinerated bottom ash, furnace bottom ash, fly ash, construction demolition waste, crushed glass, soil, rice husk ash, steel slag and compacted at pressures of above 8MPa have been reported (Forth et al 2006, Thanaya et al 2006). While this earlier work presented important initial results, further optimisation of Bitublock and its method of production were required and form the basis of the various aspects of the present invention.

The aggregate materials used in the specific embodiments described below were: steel slag (in 2002 the volume of production of metallurgical slags such as basic oxygen steel slag was 1 million tonnes; only a fifth of this was recycled); crushed glass (glass collection has increased to meet the 2006 packaging targets of 60% resulting in an excess of 300,000-400,000 tonnes of green glass) and fly ash (currently 6 million tonnes of fly ash are produced each year in the UK – only 40 to 50% of this is utilised), (Forth et al 2006).

The present application also reports the creep and moisture movement strain recorded on 7 course high by 4 brick wide single leaf Bitublock masonry structure constructed using a Class II; 1: ½: 4 ½ , cement: lime: sand mortar.

A second aspect of the present invention provides a method for producing a masonry unit, the method comprising forming a mixture containing a graded aggregate and around 6 wt % of a bituminous binder, compacting the mixture, and at least partially curing the compacted mixture.

A third aspect of the present invention provides a method for producing a masonry unit, the method comprising forming a mixture containing a graded aggregate and a bituminous binder, compacting the mixture, and at least partially curing the compacted mixture by heating the compacted mixture to a temperature of around 200 °C.

A fourth aspect of the present invention provides a masonry unit produced according to the first, second or third aspects of the present invention.

A fifth aspect of the present invention provides a masonry structure comprising a plurality of masonry units and a mortar, at least one of said masonry units comprising a graded aggregate and a bituminous binder.

Further aspects of the present invention relate to methods for producing masonry units using soil-based aggregates. In one such aspect a method comprises forming a mixture containing a soil-based aggregate and a bituminous binder, compacting the mixture at a compaction level of up to around 7 MPa, and at least partially curing the compacted mixture. In another aspect a method comprises forming a mixture containing a soil-based aggregate and at least around 13 % by weight of the aggregate of a bituminous binder, compacting the mixture, and at least partially curing the compacted mixture. In these methods employing soil or soil-based materials as the aggregates it is preferred that the mixture is compacted at a pressure of around 1 to 5 MPa, more preferably around 1 to 3 MPa, and most preferably around 2 MPa. The mixtures preferably contain around 13 to 25 % by weight of the aggregate of the bituminous binder, more preferably around 15 to 19 %, and most preferably around 18 % by weight of the aggregate of the bituminous binder. A curing temperature of around 150 to 250 °C, more preferably 180 to 220 °C, and most preferably around 200 °C may be used over any suitable time period, such as around 1 to 72 hours, more preferably around 12 to 48 hours, and most preferably around 24 hours. It should also be appreciated that soil and soil-based materials are eminently suitable for use as the aggregate or a component of the aggregate employed in any of the first to fifth aspects of the present invention.

The term "coarse aggregate" is used herein to refer to aggregate having a maximum nominal particulate size of around 14 mm and a minimum retained particulate size of

2.36 mm. The term "fine aggregate" is used herein to refer to aggregate having a minimum retained particulate size of 2.36 to 0.075 mm.

Unless otherwise stated, reference herein to 'wt %' refers to the mass of a particular material expressed as a percentage of the total mass of a mixture containing the material.

While any desirable compaction level, of up to around 7 MPa can be used, it is preferred that the compaction level is around 0.5 to 5 MPa. More preferably the compaction level is around 1 to 4 MPa. Most preferably the compaction level is around 2 MPa. Compaction may be conducted in any desirable manner, although it is preferred that the mixture of the bituminous binder and the graded aggregate is compacted within a mould, shaped to provide the mixture with the desirable shape of the final masonry unit.

In a preferred embodiment of the second, third, fourth and/or fifth aspects of the present invention, the mixture comprises up to around 10 wt % bituminous binder, more preferably around 1 to 8 wt % bituminous binder. With regard to all five of the first to fifth aspects of the present invention, it is preferred that the mixtures comprise around 3 to 7 wt % bituminous binder, and yet more preferably around 5 to 7 wt % bituminous binder. It is more preferred that the mixture comprises around 6 wt % bituminous binder.

The mixture containing the graded aggregate may include one or more types of bituminous binder in any desirable ratio. In a preferred embodiment, the mixture comprises 50 penetration grade bitumen. This type of bitumen and related relatively hard bitumens are preferred for use in applications where resistance of the final product to static loads is of relatively greater importance than curing rate. Other preferred embodiments employ a softer bitumen, such as 100 penetration grade bitumen, which are preferred for use in applications in which shorter curing times are more desirable.

Preferably the graded aggregate contains around 20 to 60 wt % of the coarse aggregate fraction. More preferably the graded aggregate contains around 30 to 50 wt

% of the coarse aggregate fraction. Most preferably the graded aggregate contains around 40 wt % of the coarse aggregate fraction.

Any appropriate coarse aggregate may be employed in the method representing the first aspect of the present invention and related aspects of the present invention. The coarse aggregate may, for example, comprise at least one of steel slag and crushed glass. By way of further example, the aggregate may be or contain soil or a soil-based material. Example 2 demonstrates the ability to employ soil as an aggregate. Further suitable aggregates and aggregate components include materials classified as 'construction demolition waste' (CDW) materials. Example 3 demonstrates the performance of a masonry unit comprising CDW material as aggregate and a bituminous binder.

It is preferred that the mixture of the graded aggregate and the bituminous binder is produced by heating and mixing the graded aggregate and the bituminous binder. Preferably said heating is effected before the graded aggregate and the bituminous binder are mixed together. At least one of the graded aggregate and the bituminous binder is preferably heated to a temperature of up to around 200 °C, and more preferably heated to a temperature of around 160 to 180 °C. It is preferred that the graded aggregate is heated to a temperature of up to around 200 °C, more preferably to a temperature of around 170 to 190 °C, and most preferably to a temperature of around 180 °C. The bituminous binder may be heated to a temperature of up to around 180 °C, more preferably to a temperature of around 150 to 170 °C, and most preferably heated to a temperature of around 160 °C. Heating of the graded aggregate and/or bituminous binder may be effected over any desirable time period. At least one of the graded aggregate and bituminous binder may be heated for a time period of up to around 5 hours, around 1 to 4 hours, or around 3 hours.

It is preferred to at least partially or fully cure the compacted mixture of graded aggregate and bituminous binder. The at least partial curing of the compacted mixture is effected by heating the compacted mixture to a temperature of up to around 250 °C, more preferably, around 80 to 240 °C, and still more preferably around 150 to 230 °C. At least partial curing may be achieved by heating the compacted mixture to a temperature of around 180 to 220 °C, more preferably around 190 to 210 °C, and most preferably around 200 °C. At least partial curing of the mixture of graded aggregate and

bituminous binder may be effected over any appropriate time period to provide an end product, i.e. a masonry unit, having the desired properties. Said at least partial curing of the compacted mixture may be effected by heating the compacted mixture for a time period of up to around 72 hours, a time period of around 1 to 48 hours, a time period of around 12 to 36 hours, and most preferably a time period of around 24 hours.

The above-defined fifth aspect of the present invention provides a masonry structure comprising a plurality of masonry units and a mortar, at least one of said masonry units comprising a graded aggregate and a bituminous binder.

Preferably the at least one of said masonry units comprises up to around 10 wt % bituminous binder, more preferably around 1 to 8 wt % bituminous binder, more preferably around 3 to 7 wt % bituminous binder, still more preferably around 5 to 7 wt % bituminous binder, and most preferably around 6 wt % bituminous binder. Any desirable bituminous binder or combination of binders may be used, although a particular suitable binder comprises 50 penetration grade bitumen.

It a preferred embodiment of the fifth aspect of the present invention the graded aggregate comprises a coarse aggregate. Preferably the graded aggregate contains around 20 to 60 wt % of the coarse aggregate, more preferably around 30 to 50 wt % of the coarse aggregate, and most preferably around 40 wt % of the coarse aggregate. Any suitable coarse aggregate may be employed, such as steel slag and/or crushed glass.

Preferably the graded aggregate employed in any of the above-defined five aspects of the present invention comprises around 80 to 40 wt % of a fine aggregate, more preferably around 70 to 30 wt % of a fine aggregate, and most preferably around 50 wt % of a fine aggregate. The fine aggregate can be chosen to suit a particular application. By way of example, the fine aggregate may comprise crushed glass.

Taking in to account the amounts of coarse and/or fine aggregates contained in the graded aggregate, the graded aggregate may comprise any appropriate amount (e.g. up to around 10 %) of a filler material, such as fly ash or the like.

The masonry structure forming the fifth aspect of the present invention may incorporate any desirable mortar. It is preferred that the mortar comprises cement, lime and/or sand. In a preferred embodiment of the fifth aspect of the present invention the mortar comprises cement, lime and sand, in any suitable ratio, such as 1 part cement : 0.5 parts lime : 4.5 parts sand, as in class II mortar. The mortar preferably exhibits a 7-day compressive strength of 5.1 MPa, a 14-day compressive strength of 9.1 MPa, and/or a 28-day compressive strength of 10.8 MPa.

The masonry structure according to the fifth aspect of the present invention preferably exhibits a 60-day creep coefficient of around 2.1, and/or exhibits shrinkage over time, for example over a time period of around 10 to 60 days.

The manufacturing process of Bitublock preferably involves three fundamental processes; mix; compact; and cure. It has been shown previously that one of the main factors affecting the properties of Bitublock is the level of compaction; it is assumed that bonding of the aggregate and the binder is achieved through encapsulation rather than chemical interaction, as is the case with cementitious bound or even clay bound units. However, although this assumption is currently being investigated qualitatively, it was decided to confirm the presence of any quantitative effects. In some clay masonry, an enlarged free expansion has been measured due to cryptoflorescence. This results from the chemical interaction of the brick and the cement mortar and can cause expansions far greater than would be expected from a consideration of the irreversible expansion of individual unbonded clay bricks. Previously, it has been proposed that any enlarged expansion or chemical interaction between the unit and the binder can be identified by the model developed by Brooks (Brooks 1990). Masonry composed of Bitublock units and cement mortar were therefore constructed and time dependent movements (creep and 'moisture' movement strains) were obtained. Composite modelling of the long-term deformations of the masonry was performed to investigate whether the encapsulation of the recycled material by the bitumen binder does eliminate any chemical interaction with the mortar.

The theory and derivation of the models has been presented elsewhere (Brooks 1990) and hence only the prediction equations are presented here.

**Modulus of Elasticity ($E_{wy}$) of Bitublock wall**

$$\frac{1}{E_{wy}} = \frac{b_y.C}{H}\left[\frac{A_w}{E_{by}.A_b + E_m.A_m}\right] + \frac{m_y.(C+1)}{H}.\frac{1}{E_m} \quad (1)$$

where: $E_{wy}$= effective modulus of Bitublock wall; $b_y$= height of block unit; C= number of courses (layer); H= height of wall; $E_{by}$= elastic modulus of block unit; $A_b$= cross sectional area of block unit; $E_m$ = elastic modulus of mortar; $A_m$= cross section area of vertical mortar joints; $m_y$= thickness of mortar bed joints; $A_w$ = cross-sectional area of masonry.

The dimensions of the Bitublock unit and wall were as follows: $b_y$ = 65mm; $A_w$ = 435x100mm; $A_b$ = 100x100mm; $A_m$= 33500mm$^2$; $m_y$= 10mm. For the 7-course wall investigated, as shown in Figure 9, equation (1) becomes:

$$\frac{1}{E_{wy}} = 0.8426\left[\frac{1}{0.229E_{by} + 0.770E_m}\right] + \frac{0.1481}{E_m} \quad (2)$$

**Creep**

To model creep behaviour, equation (3) is used however the initial modulus of the individual brick and mortar prism is replaced by an effective modulus (E') to allow for creep. Thus for a unit stress:

Creep for wall: $$C_{wy} = \frac{1}{E'_{wy}} - \frac{1}{E_{wy}} \quad (3)$$

where $E'_{wy}$ and $E_{wy}$ are the effective and initial elastic modulus of wall

**Vertical shrinkage**

The general expression of the vertical shrinkage ($S_{wy}$) at any time is

$$S_{wy} = \frac{b_y.C}{H}.S_{by} + \frac{m_y.(C+1)}{H}.S_m + \frac{b_y.C}{H}\left[\frac{S_m - S_{by}}{1 + \frac{A_b.E_{by}}{A_m.E_m}}\right] \quad (4)$$

where $S_{by}$ vertical shrinkage of block unit and $S_m$ shrinkage of mortar. For the Bitublock wall investigated, the third term of the equation is small, so that equation (4) becomes:

$$S_{wy} = 0.8426\ S_{by} + 0.1418\ S_m \quad (5)$$

The experiments described below are intended to compare the compressive strength of the Bitublock units compacted at pressures between 1 and 4 MPa with concrete block units commonly used in the UK: 3.5 – 10 MPa (Sear 2005 and British Standard-BS 6073 1981), (it has been found that the compressive strength property is a good indicator of overall block performance) and report the other physical and mechanical properties of the Bitublock units. Moreover, an investigation of the creep and 'moisture' movement strain of masonry structures, i.e. walls, constructed from the Bitublock units is described.

Embodiments of the present invention will now be described, by way of example, with reference to the accompanying figures, in which:

Figure 1 is a graph showing Bitublock grading used in comparison to a hot rolled asphalt (HRA) as specified in the British Standard 594 (BS594), an example case.schematic perspective view of an upper portion of components of an embodiment of the present invention;

Figure 2 is a graph showing Bitumen content vs. density;

Figure 3 is a graph showing Bitumen content vs. porosity;

Figure 4 is a graph showing Bitumen content vs. compressive strength;

Figure 5 is a graph showing creep test results of Bitublock units, at 1 and 2 MPa compaction levels;

Figure 6 is a graph showing expansion of Bitublock units;

Figure 7 is a photograph showing the prism creep rig;

Figure 8 is a graph showing time-dependent movements of mortar prisms;

Figure 9 is a photograph showing the loaded and control wall;

Figure 10 is a schematic presentation of the vertical and horizontal strain reading;

Figure 11 is a photograph showing the Demec gauges and strain reading;

Figure 12 is a photograph showing the steel tie-bar load cells and the data logger;

Figure 13 is a graph showing measured / predicted vertical shrinkage of control walls;

Figure 14 is a graph showing measured / predicted creep of walls;

Figure 15 shows the particle distribution of the soils used in Example 2 below;

Figure 16 shows expansion test results of the Soilblock 1 and 2 (S1 and S2) at different compaction levels (1, 2 and 4 MPa) discussed in Example 2 below;

Figure 17 shows the total strain of the Soilblock1 and 2 (S1 and S2), at different compaction level (1, 2 and 4 MPa) discussed in Example 2 below;

Figure 18 shows the creep strain of the Soilblock1 and 2 (S1 and S2), at different compaction level (1, 2 and 4 MPa) discussed in Example 2 below;

Figure 19 shows the CDW original gradation (max 10mm), and the gradation of CDW passing 2.36 mm used in Example 3;

Figure 20 shows the CDW block aggregate grading compared with the bitublock grading and the BS594, discussed in Example 3;

Figure 21. shows expansion test results for a sample tested in Example 3;

Figure 22 shows the total strain results for a sample tested in Example 3;

Figure 23 shows the creep strain results for a sample tested in Example 3;

Figure 24shows the building block aggregate grading used in comparison to hot rolled asphalt (BS594) employed in Example 4, noting UL=upper limit ; LL=lower limit;

Figure 25 shows bitumen content vs. compressive strength (cured samples) discussed in Example 4;

Figure 26 shows the expansion of the blocks tested in Example 4 at 21.0 ± 0.5 C°, and 62±2% RH;

Figure 27 shows the average movement of the samples (sp) in vertical (v) direction;

Figure 28 shows the average movement of the samples (sp) in horizontal (h) direction;

Figure 29 shows the weight change during vacuum saturation test and air drying cycles, discussed in Example 4;

Figure 30 shows the strain profile of the samples (sp), during vacuum saturation and air drying, in vertical (v) direction, discussed in Example 4;

Figure 31 shows the strain profile of the samples (sp), during vacuum saturation and air drying, in horizontal (h) direction, discussed in Example 4;

Figure 32 shows the strain profile of the samples (sp), during heating, in vertical (v) direction, discussed in Example 4;

Figure 33 shows the strain profile of the samples (sp), during heating, in horizontal (h) direction, discussed in Example 4;

Figure 34 shows the expansion on the first MA test (21 ± 1 °C and54 ± 4 % RH); discussed in Example 5;

Figure 35 shows the expansion on the second MA test (20 ± 0.5°C and 60 ± 2 % RH); discussed in Example 5;

Figure 36 shows the temperature profile of the thermal expansion test, discussed in Example 5;

Figure 37 shows the thermal expansion of the uncured sample, discussed in Example 5;

Figure 38 shows the thermal expansion of the 12 hour cured sample, discussed in Example 5;

Figure 39 shows the thermal expansion of the 24 hour cured sample, discussed in Example 5;

Figure 40 shows the weight change profile of the samples during thermal and vacuum saturation cycles (first test), discussed in Example 5;

Figure 41 shows the expansion profile of samples after the thermal and vacuum saturation cycles, discussed in Example 5;

Figure 42 shows the average results of the thermal, vacuum saturation and air drying cycles (face t, b, and s), discussed in Example 5;

Figure 43 shows typical test results of the thermal, vacuum saturation and air drying cycles (top 't' and base 'b' face), discussed in Example 5;

Figure 44 shows typical test results of the thermal, vacuum saturation and air drying cycles (side 's' face), discussed in Example 5;

Figure 45 shows weight change profile of the samples during thermal and vacuum saturation cycles (second test), discussed in Example 5;

Figure 46 shows the relationship between % increase in compressive strength of the fine mix Bitublock samples and curing regime, discussed in the Comparative Example ;

Figure 47 shows the cumulative mass loss of 0.35 mm thick bitumen samples at different curing durations for different curing temperatures, discussed in the Comparative Example ;

Figure 48 shows the relationship between compressive strength of the coarse mix Bitublock samples compacted at different compaction pressures and time of oven cure at different temperatures, discussed in the Comparative Example; and

Figure 49 shows the relation between compressive strength of the fine mix Bitublock samples and duration of cure at different curing regimes, discussed in the Comparative Example.

## EXAMPLES

### EXAMPLE 1

**Performance Criteria**

The target performance for the Bitublock unit is as follows:

- Compressive strength: ≥ 3.5 MPa at room temperature. This is in line with the compressive strength of concrete blocks commonly found in the UK: 3.5 – 10 MPa (Sear 2005 and British Standard-BS 6073 1981).
- Initial rate of suction (IRS) values shall be equal to IRS values of clay brick found in the UK (0.25-2.0 kg/m$^2$/min). The IRS is a parameter that can provide an indication of the effect of the unit on the cement mortar. Units with high IRS require very plastic mortar (high water/cement ratio), while units with lower IRS need stiffer mortar (BS3921 1985 and Vekey 2001).
- Possess specific creep (static creep strain per unit stress in MPa) of ≤ 100 microstrain, tested at 20 °C. This target is in line with the specific creep level of concrete blocks currently used in the UK (approximately 100 microstrain). The level of stress of 1 MPa shall be used for the creep test as this is considered representative in masonry experiments (Tapsir 1985 and Forth et al. 2006).

## Materials

### Bitumen Type and Content

In principal, all types of bitumen (hard/penetration grade or bitumen emulsion) can be used as a binder. However, it is preferable to use a softer grade bitumen as this requires a lower handling temperature. Also, as the samples must be cured in order to improve their resistance to long-term deformation, the use of a harder grade bitumen would not provide a significant improvement to the end product. The type of bitumen used for this investigation was 50 penetration grade bitumen (also referred to as 40/60 pen.) with a specific gravity of 1.03 and a softening point of 47 °C. A range of bitumen contents between 5 and 6.5% was considered.

| Materials | Density (gr/cm3) | Water Absorption (%) |
|---|---|---|
| Coarse aggregates (CA) | | |
| Steel slag | 3.39 | 1.9 |
| Crushed glass | 2.51 | < 0.5 |
| Fine Aggregates (FA) | | |
| Crushed glass | 2.51 | < 1 |
| Filler | | |
| Fly ash Ferrybridge | 2.16 | - |

**Table 1. The properties of the aggregate materials.**

### Aggregate type

For this investigation steel slag, crushed glass and fly ash aggregates have been used. Table 1 provides details of the aggregates used in this investigation. In order to reduce the amount of bitumen needed, (and hence enhance the economics of the mix) and yet still ensure satisfactory bitumen coating, the incorporation of waste aggregates with low absorption properties has been considered for this investigation.

### Aggregate grading

The choice of aggregate grading is largely affected by the performance criteria specified above. It has been found that a gap graded distribution of aggregates consisting of about 40 % coarse aggregates (max nominal size of 14 mm; minimum retained 2.36 mm) and 60 % fines (50 % fine aggregates (2.36-0.075mm) and 10 % filler (passing 0.075 mm) was preferable. Table 2 provides details of the aggregate and their proportions.

Figure 1 illustrates the aggregate distribution and compares it to British Standard 594, (BS594 2003). It can be seen that the fine fraction follows the lower limit of a hot rolled asphalt (HRA) grading. However, the coarser fraction (retained 2.36mm) tends towards the upper limit.

| Mix Name | Coarse aggregates: (40 %) | | Fine Agg.: 2.36-0.075mm (50 %) | Filler: < 75µm (10%) |
|---|---|---|---|---|
| | 25 % (14-10) and (10-5)mm | 15 % (5-2.36) mm | | |
| SSCF200/24 | steel slag | crushed glass | crushed glass | fly ash |

**Table 2. Type of mix and aggregate materials used**

**Bitublock unit optimisation**

The manufacture of Bitublock has been reported previously (Forth et al. 2006). Briefly, to facilitate mixing, the aggregate materials and the 50 pen bitumen were pre-heated at 160-180 °C (Withoeak 1991) for 3 hours. The loose mix was then placed in a mould and compacted. Following compaction, the Bitublock samples were cured in an oven (for this investigation the samples were cured at 200 °C for 24 hours). The performance of Bitublock is influenced by porosity and the heat curing regime. A lower porosity (higher compaction) gives improved aggregate interlock which increases the potential compressive strength. However, more efficient heat curing (higher porosity – greater depth of bitumen oxidation / hardening) improves the long-term stability of Bitublock (i.e. reduces the creep potential). In this investigation, the curing regime was fixed and the compaction level and bitumen content were varied. Figures 2 to 4 illustrate the optimisation of bitumen content and compaction level.

Referring to the aggregate grading shown in Figure 1, the minimum bitumen content for road bituminous mixtures recommended by BS594 is 6.5 % by weight of total mixture; this is to ensure adequate coating and durability. With this in mind, the bitumen content was optimised taking the figure of 6.5% as a maximum.

From Figures 2 and 3 it can be seen that a decrease in bitumen content from 6.5% to 5%, corresponds to a decrease in density and an increase in porosity. This is because the mixture becomes less workable at lower bitumen contents.

A reduction in compaction level corresponds to a decrease in density and an increase in porosity. The compressive strength trends shown in Figure 4 are in line with the trend identified for density. However, for units compacted at 2MPa there is little improvement in compressive strength beyond 6% bitumen content. Further increases in bitumen content (higher than 6.5 %) may enhance the density and hence the compressive strength. However, by observing the satisfactory degree of coating of the aggregates; the surface texture of the units and the stability of the samples during handling, together with the insignificant improvement in compressive strength of samples with over 6% bitumen content compacted at 2MPa, it was decided not to optimise the bitumen content further. For the remainder of this investigation the bitumen content was fixed at 6 % and the compaction level at 2 MPa. The compressive strength of these units still exceeded the compressive strength of concrete blocks commonly used in the UK (3.5 – 10 MPa). Also, a 0.5 % reduction in the bitumen content and a slightly higher porosity is expected to improve the long-term stability of the unit (this is considered in the next section). The initial rate of suction (IRS) of these optimised units (6 % bitumen content; 2MPa compaction level) was 0.35 kg/m$^2$.min; the 24-hr water absorption value was 5.5%.

**Long-term stability of the Bitublock units**

Prior to monitoring the time-dependent properties of the Bitublock units, the samples were stored in a controlled environment of 62% ± 1% relative humidity and 21.5°C ± 0.5°C. The samples were between 3 and 4 weeks old when they were tested. At this age, the volume stability of the unloaded samples was found to be very stable (i.e. the samples did not exhibit any expansion / shrinkage).

Figure 5 illustrates the total strain and creep of the Bitublock samples compacted at 1 and 2 MPa. The samples were loaded in a controlled environment using a static dead-weight lever arm machine (mechanical advantage of 4) providing a stress of 1 MPa. Strain measurement was performed using both a 50mm Demec gauge and electrical resistance strain (e.r.s.) gauges.

Figure 5 also illustrates the elastic strain of the samples. A summary of the elastic and time-dependent properties of the samples is shown in Table 3. Although the creep of the units compacted at both 1 and 2MPa were acceptable (in terms of their comparison

with concrete blocks), the unit compacted at 2MPa was chosen for construction in the Bitublock walls.

| Mix Name | Total Strain (με) | Elastic Strain (με) | Creep Strain [1] (με) | Elastic Modulus [2] (GPa) |
|---|---|---|---|---|
| 1 MPa compaction level | | | | |
| SSCF200/24 | 321.75 | 232.65 | 89.1 | 4.3 |
| 2 MPa compaction level | | | | |
| SSCF200/24 | 79.2 | 34.65 | 44.55 | 28.9 |
| [1] creep strain = total strain – elastic strain – shrinkage or expansion.<br>[2] elastic modulus = (1 MPa / elastic strain) | | | | |

**Table 3 Creep performance of the samples**

As mentioned above, the unloaded volume stability of the Bitublock units was obtained from units which were 3 to 4 weeks old (corresponding with the age of the creep samples). Two samples were also later manufactured and monitored from an age of 1 day (after they had cooled to room temperature). The results of these tests can be seen in Figure 6.

These results are interesting as they illustrate a behaviour similar to 'kiln fresh' clay bricks. This early age behaviour of Bitublock is currently being examined to see whether it is a consequence of water absorption / adsorption. Figure 6 illustrates how stable the Bitublock units are after 15 to 20 days.

**Mortar Details**

A class II; 1: ½: 4 ½, cement: lime: sand mortar was used throughout this investigation with 7, 14, and 28 day compressive strengths of 5.1, 9.1 and 10.8MPa, respectively. The time-dependent properties of the mortar were obtained from 75x75x200mm prisms as described previously (Brooks and Abdullah 1988), (also see Figure 7 for details of the creep rigs). Readings were taken from an age of 7 days. Prior to this the prisms were cured under plastic.

Figure 8 illustrates the creep and shrinkage of the class II mortar used in this investigation. The behaviour is similar to mortar data measured in other similar investigations. The prisms in this investigation were unsealed. Therefore, for the composite modelling exercise later, this data will have to be modified to take account of the difference in volume / surface area (v/s) ratio between the mortar prisms and the mortar in the Bitublock walls (to compensate for the different drying paths).

**Construction and testing of Bitublock walls**

The walls were constructed in a controlled environment, with temperature of: 21.5 ± 0.5 °C and relative humidity (RH) of 62±1%. The size of the Bitublock units was 100x100x65mm; the units were approximately 30 days old. Four sets of walls were constructed. Each wall was 4 units wide by seven courses high (Figure 9). The walls were constructed with a class II mortar with joints of between 5 and 8mm thick. Immediately after construction the walls were covered with plastic bags and cured for 7 days prior to being exposed to drying in the controlled environment.

Each set of two walls consisted of a loaded wall (to isolate creep) and a corresponding control wall (to obtain expansion / shrinkage deformations). On each side of the walls, vertical strain readings were taken at 4 locations and horizontal reading at three positions (Figure 10).

The strain readings were initially taken twice a day. After two weeks, the strains were recorded once a day and after one month, strains were recorded twice a week. Vertical strain readings were taken using a 150mm Demec gauge; horizontal strain readings were taken using a 200mm Demec gauge (Figure 11). Only the vertical strain data have been reported here.

The walls were loaded at an age of 7 days; a stress of 1 MPa was applied to the loaded walls. This stress was monitored and constantly maintained throughout the duration of the tests using 4 calibrated steel tie-rod load cells. (Any load adjustment required was possible by tightening / loosening the nuts on the tie-bars (Figure 12).) For practical reasons, the tests were only performed for 60 days. This was shorter than was envisaged however the data collected was considered sufficient for the purposes of this investigation.

**Modulus of Elasticity**

The average modulus of elasticity of the Bitublock walls was 4.3GPa. The elastic moduli of the unbonded Bitublock unit and the mortar prism samples were 28.9 and 2.0GPa, respectively. (The modulus of the wall is clearly influenced by the modulus of the mortar.) The measured elastic modulus compares well with the modulus predicted

using British Standard BS5628-2. For a unit strength of 14.2MPa and a class II mortar, the code predicts a modulus of 4.61GPa.

It is normal practice to apply the load to the walls within 15 minutes. According to Neville 1983, this limits the incorporation of creep within the strain measured on application of the load to acceptable levels. However, in this investigation it was reported that the time for application of the load was between 15 and 30 minutes. The elastic strain will therefore contain some creep and the elastic modulus will therefore be lower than expected. This is confirmed by the prediction of elastic modulus using the composite model (equation (2)). An elastic modulus of 5.64GPa is predicted which is higher than the measured modulus.

**Time-dependent behaviour**

Figures 13 and 14 illustrate the time-dependent behaviour of the Bitublock walls. It can be seen that the moisture movement behaviour of the Bitublock control walls is one of shrinkage (Figure 13). The Bitublock units are stable and so the movement of the wall will be controlled by the mortar which shrinks. This trend of overall shrinkage of the masonry agrees with previous findings of research performed on clay brick masonry, although this could not necessarily have been predicted and, indeed, BS5628-3 1995 recommends that all fired clay masonry expands.

Using the unbonded unit and mortar prism data (adjusted for v/s ratio differences) in the composite model expression (equation (5)) yields a slight over-prediction of shrinkage at early ages (Figure 13). However, at later ages the model under estimates the shrinkage of the walls (although the error is less than 20%, which is considered acceptable). Previously it has been shown that the composite model tends to over-predict the shrinkage of clay masonry. This is because the model does not entirely account for the interaction between the mortar and the unit in the masonry resulting from the water absorption properties of the unit. The Bitublock units in this investigation have very low water absorption properties and this interaction would be expected to have less effect. More importantly, the absence of any 'enlarged expansion' (the brickwork / brick expansion ratio is less than unity (Forth and Brooks 2000)) and the accuracy of the prediction indicates there is no chemical interaction between the elements of the masonry and that cryptoflorescence does not appear to be present.

The creep-time characteristics of the Bitublock wall can also be seen in Figure 14. The pattern of movement is similar to that seen for the mortar prisms and the unbonded Bitublock unit in Figures 8 and 5, respectively. The level of creep is also similar to that previously measured in concrete block masonry (Forth et al. 1996). Overall the composite model under-estimates the creep of the Bitublock masonry (approximately 25%) however beyond 10 days the measured and predicted creep is very similar.

The 60-day creep coefficient obtained for the Bitublock masonry of this investigation is 2.1. This value is within the range of design values suggested by BS5628-2 1995 (1.5 and 3.0 for clay and concrete block masonry, respectively). However, an exact comparison cannot be drawn as the code recommendations are ultimate values.

The investigation described above provides further proof of the concept of Bitublock and that the physical and mechanical properties of Bitublock units are at least equivalent to concrete block masonry units used in the UK.

This is the first time the elastic and time-dependent properties of Bitublock masonry have been investigated. For the experimental conditions of this investigation, composite modeling using un-bonded unit and mortar deformations gives reasonable estimates of elastic modulus, shrinkage and creep of Bitublock masonry. The use of composite modeling has helped to confirm the apparent absence of any chemical interaction between the mortar and the unit in terms of what effect this might have on the long-term behaviour of Bitublock masonry. The manufacture of Bitublock is an encapsulation process (its properties are not dependent on chemical binding). The results of this investigation help to confirm that the recycled material used in Bitublock is adequately encapsulated by the bitumen.

Overall, Bitublock is compliant with the design suggestions of the relevant British Standards for masonry.

## EXAMPLE 2

The objective of this Example was to investigate and evaluate the engineering performance (compressive strength and creep) of masonry units comprising soil as the

aggregate with a bitumen binder ("Soilblock") and compare these with the properties of coarse aggregate concrete blocks found in the UK.

The soil used in this investigation was from a construction site in Hunslet, Leeds, UK. The soil was sieved through a 2.36mm sieve size. Two types of soils were used. The particle sizes of the soils were of a relatively continuous grading (see Figure 15), where Soil 2 was slightly finer than Soil 1. The soil fraction was very brittle and could easily be crushed by hand. The larger particles were porous by nature.

| Properties | Soil 1 | Soil 2 |
|---|---|---|
| Liquid limit | 29 % | 27 % |
| Plastic limit | 23 % | 22 % |
| Plasticity index | 6 % | 5 % |
| Specific gravity | 2.397 | 2.400 |
| Water absorption | 12.1 % | 11.2 % |

**Table 4. Properties of the soils.**

By referring to the data in Table 4 both Soil 1 and Soil 2 have a liquid limit less than 50% (i.e., low plasticity). These are the preferred types of soils to be used for producing Soilblock. Utilization of soils with high plasticity or expansive soils for most works can affect the volume stability when subjected to moisture. Soil 1 falls into category 3 (inorganic silt with low compressibility); Soil 2 can be grouped between category 2 and 3 (between inorganic silt with low compressibility and inorganic clays with low plasticity).

The bitumen used was 100 penetration grade and with specific gravity (SG) of 1.02. The bitumen content was determined based on the minimum content required for satisfactory coating. The Soilblocks were produced as follows. The soil (passing 2.36mm) and bitumen (pre heated at 160°C for 3 hours) were mixed to produce Soilblocks. The compaction levels investigated were 1, 2, and 4 MPa. The samples were compacted at 100-110°C. Following compaction, the Soilblock samples were cured in an oven. When using a 50pen bitumen and cured at 160°C, the curing duration required to satisfy creep performance was 72 hours; softer grade bitumen (i.e. 100 pen grade bitumen) was found to undergo more effective curing. In order to reduce

the curing duration in this investigation, the samples were cured at 200°C for 24 hours. The volumetric and mechanical properties of the samples are given in Table 5.

**Table 5. Properties of the Soil Bitublock samples**

| Compaction level (MPa) | IRS* (kg/ mm.min) | Density (gr/cm3) | $S_{Gmix}$ | Poro-sity (%) | Uncured comp. strength (MPa) | Cured ** comp. strength (MPa) | Water Abs. *** (%) |
|---|---|---|---|---|---|---|---|
| Soilblock 1 | | | | | | | |
| 1 MPa | 0.08 | 1.478 | 1.993 | 25.9 | 0.75 | 4.7 | 5.4 |
| 2 MPa | 0.05 | 1.595 | 1.993 | 20.0 | 1.5 | 9.1 | 4.8 |
| 4 MPa | 0.09 | 1.671 | 1.993 | 16.1 | 2.2 | 12.2 | 4.2 |
| Soilblock 2 | | | | | | | |
| 1 MPa | 0.09 | 1.474 | 1.995 | 26.10 | 0.62 | 3.6 | 5.8 |
| 2 MPa | 0.07 | 1.566 | 1.995 | 21.5 | 1.3 | 7.6 | 5.1 |
| 4 MPa | 0.10 | 1.648 | 1.995 | 17.4 | 2.0 | 10.8 | 4.45 |

* initial rate of suction (IRS) ** 200°C for 24 hours ***24 hours water immersion

The porosity of the samples was relatively high compared to Bitublock samples previously made with waste aggregate materials, e.g. as in Example 1 above. The reasonably low water absorption values suggest that the soil agglomerations were encapsulated successfully by the bitumen.

The initial rate of suction (IRS) value of the Soilblock was found to be lower than the range of IRS values for clay bricks recommended in the UK (between 0.25-2.0 $kg/m^2/min$). It is thought that the low value of IRS recorded in this investigation is due to the surface of the samples which was smooth and mostly coated by a thin bitumen film that has hydrophobic character. This suggests that stiffer mortars are more suitable for producing Soilblock masonry. This is advantageous in a sense that in remote geographical areas where quality control may be low and the availability of water restricted, a poor quality, stiffer mortar could potentially be tolerated.

The compressive strengths of the uncured samples were considered borderline acceptable for the UK market however they are suitable for low-rise construction in developing / transitional countries. The strengths significantly increased when cured. The compressive strength of the cured samples complies with the British Standards for concrete masonry units, where the range of strength expected is between 2.8 - 10

MPa. This indicates that the curing regime applied (200 °C for 24 hours) gave satisfactory results. The mode of failure of the samples was brittle.

Volume stability was carried out by monitoring expansion or shrinkage of the units. The samples were placed on a table at room temperature of 21±1 °C and relative humidity (RH) of 60±5 %. Monitoring began immediately after manufacture (once the blocks had cooled down). It was found that initially the samples expanded with time however beyond two weeks they were stable (see Figure 16). The results suggested that the expansion was caused by moisture adsorption from the environment. Lower expansion occurred on samples with higher compaction level (see Figure 16).

The magnitudes of the expansions at the end of the test are given in Table 6 in accordance with the results in Figure 17.

| Compaction level | Exp. of Soilblock1 (microstrain) | Exp. of Soilblock2 (microstrain) |
|---|---|---|
| 1 MPa | 594.0 | 650.9 |
| 2 MPa | 509.85 | 551.9 |
| 4 MPa | 396.0 | 475.8 |

**Table 6. The magnitude of the Soilblock expansion at the end of test.**

It was observed that the expansion of the samples did not cause any visible cracks. The compressive strengths (Table 5) were obtained after the expansion tests were completed. This indicated that despite the expansion, the samples still gave acceptable strength. If the Soilblocks are used to construct a wall, the expansion of the unit may counteract some or all of the shrinkage that commonly occurs in cement mortar. The expansion of the unit could also provide a pre-stressed condition to the wall structure that can improve the ability of the wall to bear horizontal loads.

For the creep test, the samples were loaded using a static dead-weight lever arm machine with mechanical advantage of 4. The stress applied was 1 MPa. The strain was monitored on each of the four faces of each sample and measured by means of a 50mm Demec gauge, and then averaged. The creep performances of the samples under a 1 MPa stress are shown in Figures 17 and 18. A summary of the results is also given in Table 7.

**Table 7. Creep performance of the Soil Bitublock samples.**

| Comp level (MPa) | Total Strain (με) | Elastic Strain (με) | Elastic modulus (MPa) | Creep Strain [1] (με) | Expansion at creep test * (με) |
|---|---|---|---|---|---|
| Soilblock1 | | | | | |
| 1 | 320.80 | 170.50 | 5865.10 | 150.3 | * |
| 2 | 160.40 | 70.55 | 28348.68 | 89.85 | * |
| 4 | 115.20 | 45.60 | 87719.29 | 69.60 | * |
| Soilblock2 | | | | | |
| 1 | 380.60 | 200.21 | 4994.75 | 180.39 | * |
| 2 | 185.50 | 90.25 | 22160.66 | 95.25 | * |
| 4 | 145.20 | 60.32 | 66312.99 | 84.88 | * |

[1] creep strain = total strain – elastic strain – shrinkage or expansion.

* the samples were tested for creep after the expansion was stable (at zero expansion).

Figures 17 and 18 indicate that creep had finished after 2 days. The total creep of the samples compacted at 2 and 4 MPa were less than 100 microstrain and are at least compatible to creep recorded in concrete blocks. The level of creep and the cessation of creep after such a short time (as well as the compressive strength failure mode) confirm the manufacturing process has converted the bitumen bound unit from a viscous-plastic material to an elastic brittle material. Realistically, for low rise construction in developing countries, a higher level of creep may be acceptable. As such, the curing regime (temperature and duration) adopted for this investigation could therefore be reduced.

From Example 2 it can be concluded that soil can be used as an aggregate for a masonry block, which can possess compressive strengths comparable to concrete blocks commonly used in the United Kingdom. Expansion can be reduced by increasing the compaction level. A curing regime of 200°C for 24 hours was found sufficient to harden the bitumen and hence meet the required Soilblock performance. However, this regime could be relaxed for developing country construction. The creep of the Soilblock can be reduced by increasing compaction level, but a minimum compaction level of around 2 MPa is currently recommended.

**EXAMPLE 3**

The objective of this Example was to investigate and evaluate the engineering performance (compressive strength and creep) of masonry units comprising construction demolition waste (CDW) as the aggregate with a bitumen binder ("CDWblock") and compare these with the properties of coarse aggregate bitublocks prepared in accordance with Example 1.

Construction demolition waste (CDW) is a non-homogeneous material as it may come from demolition of various types of buildings or constructions. The CDW used for this investigation consisted of a mixture of broken concrete, clay brick masonry, sand cement mortar, and reclaimed asphalt pavement (RAP) in a random proportion. The CDW particle size distribution is shown in Figure 19, with maximum particle size of 10mm. The bitumen used as the binder was 100pen grade bitumen. This is relatively soft grade bitumen.

The gradation of the CDW used was found to be of a relatively continuous grading. It contains a filler component (particle sizes passing 0.075mm) of 2 % by weight of total aggregates, or 3.5 % of the fine aggregates (passing 2.36 mm). The properties of the CDW aggregates were tested in line wit the BS 812 (1995), and are given in Table 8.

| Properties | Unit | Coarse CDW (> 2.36mm) | Fine CDW (< 2.36mm) |
|---|---|---|---|
| Density (bulk) | grm/cm3 | 2.415 | 2.341 |
| Density (ssd) | grm/cm3 | 2.478 | 2.433 |
| Density (app) | grm/cm3 | 2.569 | 2.578 |
| Water absorption | % | 2.5 | 3.9 |

**Table 8. Properties of the CDW aggregates.**

The properties the CDW aggregates were found very comparable with commonly aggregates available for building industries. However, as the CDW is processed from waste materials, therefore the homogeneity of the aggregate component may not be consistent.

The CDW material particles sizes were initially graded (sieved) into: a coarse fraction of 10-5mm and 5-2.36mm, and a fine fraction of all passing 2.36 mm. Initially the aggregate grading used for the CDWblock was the same as a Bitublock (see Example

1), but with maximum particle size of 10mm. The aggregate composition was: 40% coarse fraction, 50 % fine fraction, and 10 % coal fly ash filler as shown in Figure 20 which is completed with a hot rolled asphalt (HRA) grading of the BS 594, for comparison.

The CDWblocks were produced with the lowest bitumen content possible that can give adequate bitumen coating with satisfactory stability during handling and satisfactory performances when compacted at low compaction effort. Bitumen content of 5 % by weight of total mixture was initially tried. The static compaction effort was 1 MPa for 1 minute. The degree of bitumen coating was satisfactory; however the shape of the blocks was not stable. In order to improve the CDWblock shape stability, the aggregate grading was slightly modified, by reducing the filler content from 10 % to 5%, but increasing the fine fraction from 50 % to 55 %, where the coarse fraction remain at 40 % (Figure 20). The proportion of the fine fraction was adjusted in order to maintain the filler content of the mix to remain at 5 %, where the added filler used was coal fly ash.

In order to reduce water absorption, the bitumen content was increase from 5 % to 5.5 %. The performances of the newly produced samples were satisfactory, with surface texture slightly more open, but with better bitumen coating, and theoretically with a thicker bitumen film. A CDWblock with modified gradation (Mix B in Table 2) was then produced with compaction effort of 1, 2, and 4 MPa, with bitumen content of 5.5 % by weight of total mixture. In this investigation the samples were cured at 200°C for 24 hours.

The IRS values of the CDWblock were found somewhat lower than the range of IRS values for clay brick found in the United Kingdom (between 0.25-2.0 kg/m$^2$/min). Low IRS values were obtained because the aggregates were evenly coated by bitumen which has hydrophobic character. This suggested that the CDWblock tested in this experiment might be amenable for application with a relatively stiff mortar.

The properties of the samples are given in Table 9.

**Table 9. The Properties of the CDWblocks.**

| Mix | Comp. effort (MPa) | Density ($g/cm^3$) | Porosity (%) | IRS (kg/ m2.min) | Water Abs * (%) | Comp. Strength (MPa) | |
|---|---|---|---|---|---|---|---|
| | | | | | | uncured | cured |
| Mix A | 1 | 1.925 | 15.1 | 0.105 | 6.9 | 2.0 | 8.1 |
| Mix B | 1 | 1.872 | 17.5 | 0.028 | 3.3 | 2.8 | 10.2 |
| | 2 | 1.950 | 14.0 | 0.021 | 2.7 | 3.8 | 17.5 |
| | 4 | 1.992 | 12.2 | 0.018 | 1.6 | 5.4 | 25.8 |

* 24 hours immersion in water

The performance of Mix A was affected by its grading which contained 10% filler compared with Mix B with 5 % filler content, and also affected by its lower bitumen content. As Mix A was finer than Mix B, at a 1 MPa compaction level, Mix A was of lower porosity than Mix B. However the water absorption was higher as the bitumen film was thinner. The compressive strength of Mix B was better than Mix A, as Mix B was slightly coarser and of thicker bitumen film. The performance of Mix B was improved at higher compaction levels. The compressive strength of the samples well exceeded 7 MPa, a common compressive strength value for concrete blocks found the UK.

The water absorption of Mix B was reasonable, which indicated that at 5.5 % bitumen content, the samples became more impermeable. The IRS values were found relatively lower than the typical IRS values of clay brick found in the UK (0.25 – 2.0 kg/m2/min). This suggests that it might be appropriate to use CDWblocks with a sand cement mortar with a relatively lower water cement ratio.

The mode of failure of the samples under compressive strength test was generally brittle. The failure mode of the uncured samples was slightly more plastic than the cured one. This may be because the uncured bitumen contained more viscous component than the cured one.

Before doing creep tests, the cured samples were tested for their expansion at room environment condition (21±0.5 ℃ and 46 % relative humidity (RH)). It was found that the samples expanded then stabilized after about 7 days. The samples with higher compaction level gave lower expansion as shown in Figure 21.

It was also observed that the samples did not crack which indicates that the expansion was not excessive. The expansion of the unit would be neutralized by the shrinkage of the sand cement mortar joints in wall construction. The expansion can also give a pre-stressed condition to the wall structure which can improve the ability of the wall to receive horizontal load.

The samples were then tested for creep. The stress applied was 1 MPa. This stress is commonly applied in masonry experiments in order to evaluate specific creep, i.e. creep strain per MPa unit stress. The creep test results are shown in Figures 22 and 23, and summarized in Table 10.

**Table 10. Creep performance of the CDWblock samples.**

| Comp level (MPa) | Total Strain (με) | Elastic Strain (με) | Creep Strain [1] (με) | Expansion at creep test * (με) |
|---|---|---|---|---|
| 1 | 173.25 | 54.45 | 118.8 | * |
| 2 | 143.55 | 49.5 | 94.05 | * |
| 4 | 113.85 | 34.65 | 79.20 | * |

[1] creep strain = total strain – elastic strain – shrinkage or expansion.

* the samples were tested for creep after the expansion stabled (at zero expansion).

All of the samples gave creep strain at least equal to a concrete block commonly used in the UK. In order to ensure a better deformation resistant, a creep strain of less than 100 microstrain is recommended, therefore a compaction level of at least 2 MPa is suggested.

This section is intended to compare the CDWblock with Bitublock (see Example 1). A comparison of data relating to the two samples is presented in Table 11.

| Description | CDWblock (Mix B) | Bitublock |
|---|---|---|
| Max aggregate size | 10mm | 14mm |
| Coarse agg. | CDW | steel slag |
| Fine agg. | CDW | crushed glass |
| Filler | coal fly ash | coal fly ash |
| Filler content | 5% | 10% |
| Bitumen grade | 100pen | 50pen |
| Bitumen content | 5.5 % | 6 % |
| Bitumen film thickness (BFT) | 6.9 micron | 6.2 micron |

| | | |
|---|---|---|
| Compaction level | 2 MPa | 2 MPa |
| SG of bitumen | 1.02 | 1.03 |
| Curing regime | 200°C for 24 hr | 200°C for 24 hr |
| Porosity | 14% | 17.4 |
| Compressive strength (cured) | 25.9 MPa | 14.2 MPa |
| Specific creep | 94.1 microstrain | 44.6 microstrain |

**Table 11. Data comparison between the CDWblock and the Bitublock.**

The aggregate surface area was calculated by referring to the method recommended by the Asphalt Institute. The results are summarized in Table 12.

**Table 12. Calculation of aggregate surface area (ASA), (Asphalt Institute, 1984).**

| Sieve size | | Estimated cum pass (%) * | | Surface area factor (SAF) | Agg. surface area (ASA) (m2/kg) | |
|---|---|---|---|---|---|---|
| Inch / No. | mm | CDWblock | Bitublock | | CDWblock | Bitublock |
| | | a | | b | c = a x b | |
| ¾ " | 19.0 | 100 | 100 | 0.41 | 0.4100 | 0.4100 |
| 3/8 " | 9.5 | - | - | | | |
| No. 4 | 4.75 | 74 | 74 | 0.41 | 0.3034 | 0.3034 |
| No. 8 | 2.36 | 60 | 60 | 0.82 | 0.4920 | 0.4920 |
| No. 16 | 1.18 | 52.5 | 52.5 | 1.64 | 0.8610 | 0.8610 |
| No. 30 | 600 µm | 46 | 46 | 2.87 | 1.3202 | 1.3202 |
| No. 50 | 300 µm | 25 | 25 | 6.14 | 1.5350 | 1.5350 |
| No. 100 | 150 µm | 14 | 14 | 12.29 | 1.7206 | 1.7206 |
| No. 200 | 75 µm | 5 | 10 | 32.77 | 1.6385 | 3.2770 |
| ASA (sum) | | | | | 8.2807 | 9.9192 |

* estimated based on the aggregate grading shown in Figure 3.

The bitumen film thickness of the CDWblock and Bitublock was in line with the aggregate grading, aggregate surface area, and the bitumen content. All of the samples showed a good degree of coating. The porosity of the CDWblock was about 3 % lower than the Bitublock. This was attributed to the use of softer bitumen grade for the CDWblock, where at the same compaction level and correct temperature, the CDWblock would be more workable during compaction. The effect of this lower porosity was revealed on the higher compressive strength of the CDWblock due to better aggregate interlock. In addition to lower porosity, the materials used for the CDWblock would have given better aggregate friction compared to the large portion of crushed glass used for the Bitublock. The results also revealed that the utilization of

softer binder was effective in line with the curing regime applied. The specific creep results were principally satisfactory (less than 100 microstrain). The Bitublock performed better than the CDWblock. This situation suggests that the harder binder gave better resistant to static load as.

Some conclusion can be withdrawn from the investigation were as follows. The CDW materials were found very suitable for producing bitumen bound masonry block (CDWblock). A compaction level minimum of 2 MPa and the curing regime applied (200 °C for 24 hours) were found to give satisfactory performances (compressive strength and creep). Utilization of softer binder was found effective in line with the curing regime applied. The performances of the CDWblocks were found to be at least equal to the concrete blocks commonly used in the UK.

**EXAMPLE 4**

In this investigation a bitublock masonry unit was produced incorporating steel slag, crushed glass and coal fly ash as the aggregate. The material proportions are given in Figure 24. The properties of the materials selected are shown in Table 13.

| Materials | Density (gr/cm3) | Water Abs (%) |
|---|---|---|
| Coarse agg (CA); > 2.36mm | | |
| Steel slag | 3.39 | 1.9 |
| Crushed glass | 2.51 | < 0.5 |
| Fine agg. (FA); (2.36-0.075mm) | | |
| Crushed glass | 2.51 | < 1 |
| Filler; passing 0.075mm | | |
| Fly ash Ferrybridge | 2.16 | - |

**Table 13. The properties of the aggregate materials.**

In principal all types of bitumen (hard/penetration grade or bitumen emulsion) can be used as a binder. However, it is preferable to use softer grade bitumen as this requires a lower handling temperature. The type of bitumen used for this investigation was 50 penetration grade (50 pen or 40/60 bitumen) with a specific gravity of 1.03 and a softening point of 47 °C.

The blocks were produced using a hot mix method. The aggregate materials that had been proportioned and the 50pen bitumen were pre-heated at 160-180 °C for 3 hours. The loose mix was then placed in a mould and compacted. The sizes of the samples were 100x100x65mm. The samples were cured in an oven at 200°C for 24 hours. In this investigation the compaction level applied from a vertical direction was 1, 2 and 4 MPa, and the bitumen contents were varied from 5 to 6.5%, with 0.5 % increments.

Considering the compressive strength results shown in Figure 25, a compaction level of 2 MPa and bitumen content of 6 % were chosen, as they had given satisfactory results, i.e. adequate degree of coating, stable during handling and sufficient cured compressive strength. Additional bitumen content was not found to give significant improvement. The compressive strength of the units at this option was 14.2 MPa which still exceeded the compressive strength of concrete blocks commonly used in the UK: 2.8 – 10 MPa (BS 6073-1, 1981). The creep strain per MPa stress (specific creep) was also satisfactory (Table 3), i.e. less than 100 microstrain.

The properties tested were volumetric properties (density and porosity), initial rate of suction (IRS), water absorption, compressive strength. A summary of test results at the chosen 6% bitumen content and 2 MPa compaction effort is given in Table 14.

**Table 14. The properties of the Building Blocks at 6% bitumen content, compacted at 2 MPa.**

| Properties | Unit | Value |
|---|---|---|
| Density | $g/cm^3$ | 2.044 |
| Porosity | (%) | 17.4 |
| IRS | kg/m2.min | 0.35 |
| Water abs. by 24 hr immersion | (%) | 5.5 |
| Comp. Strength<br>- Uncured<br>- Cured * | MPa | <br>2.6<br>14.2 |
| Specific creep | microstrain | 44.6 |

* cured at 200° C for 24 hours

The IRS values of the blocks were found to be at a lower range compared to IRS values for clay brick found in the United Kingdom (between 0.25-2.0 $kg/m^2/min$). This suggests that the bitublocks tested in this experiment may optimally be employed with a relatively stiff mortar.

Two samples with size of 100x100x65mm were tested. The expansion of the samples was monitored by means of a 50mm Demec gauge. The test results are shown in Figure 26 (taken after a 24 hour heat curing cycle and a 24 hour cooling down period). On the first test, two samples were conditioned at room environment, i.e. at temperature (21. 0 ± 0.5 C°) and 62±2% relative humidity (RH). The strain measured was on the vertical direction only. It was found that the samples expanded with average value of about 180 microstrain ($10^{-6}$) as shown in Figure 26.

In order to evaluate the performance of the samples at different relative humidity, the second test was done by initially conditioning two samples at room environment. The temperature was relatively constant at 21.0 ± 0.5 C°, but the humidity fluctuated at 62±2% RH. The samples were also conditioned at 12%RH and 85%RH which were carried out by using desiccators filled with lithium chloride and potassium chloride hygrostatic solution respectively where two samples were tested for each conditioning.

The expansion reading was taken in vertical and horizontal direction at certain time interval until the expansion stabilized. Then the conditioning was changed. The results are shown in Figures 27 and 28. The samples were initially left overnight at room environment with 62±2% RH before the first strain reading was noted. The samples were then conditioned in a desiccator with 12%RH. The samples gradually shrunk then stabilized at 100 microstrain. Starting on day 11th, the samples were taken out from the desiccator and left at room environment (62±2% RH). The samples slowly expanded then stabilized at 20 microstrain. Starting from day 24$^{th}$ the samples were put into a desiccator with 85%RH. The samples expanded most then stable at 340 microstrain. Starting for day 44$^{th}$ and then the following days, the samples were consecutively condition until stabilized at room environment, then at again at 12%RH.

The second test confirmed that the volume stability of the samples was affected by changes in relative humidity. Conditioning to lower relative humidity caused the samples to shrink and vice versa. However, the magnitude of expansion and/or shrinkage was found not proportional to the changes in RH. The results indicated that the samples movement were partly reversible and irreversible. This situation is similar to clay brick.

The next test was carried out in three cycles. Each cycle consisted of the following procedure: the samples were initially vacuumed without water for 30 minutes, then water was supplied until the samples fully immersed. The immersed samples were then vacuumed for a further 30 minutes, after that they were left soaked for 30 more minutes. Then the samples were weighed and air dried at room environment until the volume of the sample stabilized. Measurement of the volume stability (expansion or shrinkage) and weighing of the samples was done every 24 hours. The results are shown in Figures 29, 30 and 31.

Figure 29 shows that the vacuum saturation test applied gave 6 % water absorption. After the test, moisture loss occurred during air drying of the samples. Referring to Figure 30, within the first cycle, vacuum saturation caused the samples to expand to about 500 microstrain, then samples were air dried at room environment. After 1 day of air drying the samples continued to expand to about 1600 micostrain. Expansion still occurred on the following two days to a maximum of 1850 microstrain, regardless of continuous moisture loss as shown in Figure 29. The samples were then very gradually shrunk before becoming stable at 1000 microstrain. It took more than three weeks for the samples to stabilize on the first cycle. Then two more vacuum saturation and air drying cycles were carried out. The room temperature during air drying was relatively constant at 21.0 ± 0.5 C°, but the relative humidity (RH) fluctuated. During the first cycle of air drying the RH was 62±2 %, then 50±2 % on the next two cycles. This should have affected the rate of expansion and shrinkage of the samples. Figure 31, revealed a similar pattern in the horizontal direction, but of smaller magnitude than in vertical direction (Figure 30). The results indicated that samples movements were un-isotropic, where the horizontal movements were about 14 % less than the vertical movements. This may be associated with the vertical direction of compaction.

Figure 30 and 31 show that the expansion due to vacuum saturation on the first cycle was totally irreversible. Then partial reversible movement occurred during air drying, i.e. the samples expanded then shrunk, but did not return to their position at the start of air drying conditioning. The results indicated that due to moisture exposure the volume stability of the Bitublock were largely (partly) irreversible from its original condition. This property is similar to concrete masonry (CST, 2007), as well as to clay brick. The moisture expansion of the Bitublocks was found comparable to the expansion of fired clay brick which can vary between 500-2500 microstrain.

The results also suggest that the volume stabilities of the Bitublocks were in line with the mechanism of shrinkage and swelling in concrete technology.

A thermal expansion test was carried out by conditioning the samples in an oven at 70 °C for 3 hours. This time was sufficient to generate the targeted heat on the core of the samples (tested using a thermocouple inserted into one of the samples). After heating, the samples were left at room environment (21.0 ± 0.5 °C, with 50±2% RH) until stabilized. The results are presented in Figures 32 and 33.

Referring to Figure 32 (strain in vertical direction), within the first heating cycle the samples expanded to 750 microstrain ($10^{-6}$). Then the samples were taken out from the oven and conditioned at room environment for 2 days during. Within the fist day at room environment the samples shrunk almost to its original position and then slightly expanded on the next day (as had been experienced). Similar procedures were carried out on the next two cycles. Strain in the horizontal direction was of a similar pattern, but with lower strain magnitude as shown in Figure 33. There was an element of un-isotropic movement between the vertical and horizontal movement (similar to the movement due to vacuum saturation and air drying. The horizontal movement was about 13 % less than the vertical one.

Thermal expansion of the blocks was found highly reversible, similar to concrete masonry. The coefficient of thermal expansion on the Building Blocks was around 600-700 microstrain or ($10^{-6}$) per 70 °C, or about 8.6-10x$10^{-6}$ /°C. This coefficient should have affected by the size of the samples (100x100x65mm) and the level of curing regime applied. The coefficient is comparable to the coefficients of expansion of concrete masonry units, i.e. 7.2 to 9.0 x $10^{-6}$ /°C.

From this investigation the following conclusions can be made. The performance of the bitublocks, in term of compressive strength, were comparable to concrete blocks currently used in the UK. The expansion of the bitublocks was affected by environmental relative humidity (RH). Conditioning at lower relative humidity caused the samples to shrink and vice versa. The magnitude of expansion and/or shrinkage was found not proportional to the changes in RH. Due to moisture exposure the bitublocks were generally expanded, and the volume stability of the bitublocks was

found largely irreversible from its original condition. The moisture expansion of the bitublocks was comparable to clay bricks. Thermal expansion of the bitublocks was found highly reversible, with coefficient of thermal expansion comparable to concrete masonry block.

**EXAMPLE 5**

In this investigation a bitublock masonry unit was produced incorporating steel slag, crushed glass and coal fly ash as the aggregate. The properties of the materials selected are shown in Table 15. The material proportion which was based on modification of aggregate grading for hot rolled asphalt (BS594) is shown in Table 16.

| Materials | Density (gr/cm3) | Water Absorption (%) |
|---|---|---|
| Coarse aggregates (CA) | | |
| Steel slag | 3.39 | 1.9 |
| Crushed glass | 2.51 | < 0.5 |
| Fine Aggregates (FA) | | |
| Crushed glass | 2.51 | < 1 |
| Filler | | |
| Fly ash Ferrybridge | 2.16 | - |

**Table 15 The properties of the aggregate materials**

| Aggregates | % | Materials |
|---|---|---|
| Coarse aggregates: | | |
| - (14-10)mm | 5 | steel slag |
| - (10-5)mm | 20 | crushed glass |
| - (5-2.36)mm | 15 | crushed glass |
| Fine aggregates: | | |
| - (2.36-0.075)mm | 50 | crushed glass |
| Filler: | | |
| - passing 0.075mm | 10 | coal fly ash |

**Table 16. Proportion of aggregate used.**

The type of bitumen used for this investigation was the same as in Example 4, i.e. 50 penetration grade (50 pen or 40/60 bitumen) with a specific gravity of 1.03 and a softening point of 47 °C. The samples were produced as explained above in Example 4 and consequently possessed the same properties as the bitublocks employed in Example 4.

The cured compressive strength was 14.2 MPa, which well exceeds the range of the compressive strength of concrete block recommended by the British Standard, i.e. 2.8-10 MPa (BS6073-1, 1981). The creep strain per MPa stress (specific creep) was also satisfactory, i.e. less than 100 microstrain.

Inspired by the intention for knowing the effect of different heat curing regime applied on the expansion of the samples, thinner samples were produced with size 100x100x35 mm. With thinner samples, more severe heat curing effect to the samples would occur. Two different types of investigations carried out. The difference was on the way the thin samples cured and the curing regime applied. On the first MA test the samples were cured in oven in vertical position. The curing regime was: uncured, 12 hours and 24 hours cured at 200 °C. After heat curing, a pair of Demec point was pasted on the samples by means of a super glue (on each of the four vertical faces only). The second MA test was carried out after obtaining results from the first test (described on the next part of this section), where the samples were cured in oven for 24 hours at 200 °C. The strain was measured using a 50mm Demec gauge.

The results of the first MA test are presented in Figure 34. It is shown that the expansion and time required to stabilize are in line with the curing regime. The uncured sample underwent lowest expansion and stabilized fastest, and vice versa. The samples heat cured for 24 hours required about 9 days to stabilize. The results suggested that the expansion was due to moisture absorption from the environment. The sample with longer (more intense) curing regime would be drier hence adsorbed more moisture, therefore gave higher expansion and require longer time to become stable.

In order to confirm the results from the first test, a second test was carried out with different sample positioning during heat curing. Three samples were tested. The samples were placed on a metal tray during heat curing. The top (upper) side or face of the samples during heat curing was labelled 't', in x and y strain measurement. The base (lower/opposite side of face t) that was placed facing a metal tray was labelled as 'b'. The side faces of the samples were labelled as 's' 1 to 4.

The MA test results (average of three samples) from the second test are shown in Figure 35. Face t gave highest expansion followed by face s. Face b was the lowest.

As face t was on the top/upper side of the samples during heat curing, therefore it totally exposed to heat and had the best access for the evaporation of the volatile components of the sample (bitumen), then followed by face s then face b. So face t underwent most effective heat curing. The results was found in line with the results from the first MA test as shown in Figure 34, where the samples cured longest (24 hours) gave highest expansion. The magnitude of the expansion was affected by the fluctuation on the relative humidity, but the trend was similar. On the samples cured for 24 hours, the results in Figure 34 were slightly lower than the in Figure 35, as the relative humidity was lower.

The three samples: uncured, 12 hours cured and 24 hours cured at 200 °C, were then subjected to a thermal expansion test at 70 °C as described within the following sections.

The expansion was monitored using electric resistant strain (ers) gauges. Before pasting the ers gauges, a thin layer mixture of chemical plastic padding glue paste and hardener was applied onto certain area of the sample. The glue paste was left to dry for 15-20 minutes, then smoothened by using a piece of sand paper. After that the smoothened surfaced was cleaned with a cleaning solvent, then the ers gauges were stick (on four faces of the samples in vertical direction) by means of a transparent super glue. Into one of the three samples, a thermocouple was inserted to monitor the temperature within the core of a sample. Then electric cable was welded to the ers gauges then connected to an Orion data logger, and the samples were put into an air circulated and temperature controlled oven.

The test was run for three cycles using a timer. The heating time was set for two and a half hours. This time was found sufficient to reach a temperature of 70 °C on the core of the sample. The cooling time was set for 6 hours to ensure sufficient time to achieve room temperature as the door of the oven was kept closed. The strain data was electronically and automatically recorded every 15 minutes. The temperature profile during the test is shown in Figure 36. The thermal expansion test results of the uncured, 12 hours cured and 24 hour cured samples are presented in Figures 37, 38 and 39 respectively.

Figure 37 shows that the ers gauge on face 4 (f4) of the sample gave a very different results, hence discarded. When the rest of the data are averaged, the expansion is around 1400 micro strain. There is only a slight reduction in expansion on the following cycles. Results in Figure 38, also gave a strange result for the ers gauge on face 4 (f4). The rest of the data gave an average of 400 microstrain expansion.

All ers gauges on the test results as shown in Figure 39, gave a reasonable trend with result on face1 (f1) much smaller than others. If the rest of the results are averaged, the expansion was about 220 microstrain. Figures 37 to 39 indicate that the samples with more intense heat curing regime, gave lower thermal expansion.

There was an indication that the later thermal expansion cycle, gave a small or no significant changes on their expansion. This is because the samples had been previously subjected to intense heat curing, hence had become more elastic where the viscous component of the bitumen had been reduced due to volatilization and oxidation.

Figure 39 revealed that the coefficient of thermal expansion of the 24 hour cured samples was between 180-200 microstrain ($10^{-6}$) per 70 °C or about 2.6-2.9 x$10^{-6}$/ °C, which is lower than the coefficient of thermal expansion of clay bricks: 4.5 to 7.2 x $10^{-6}$/°C, and of concrete masonry unit: 7.2 to 9.0 x $10^{-6}$/°C. This means that the bitublock would give a relatively lower expansion (more stable).

Expansion measurements of the samples were carried out using a 50mm Demec gauge as follows:

a. Initially at room temperature (21 ± 1 °C and relative humidity (RH) of 54 ± 4 %).
b. After heating at 110 ± 5 °C for 2 hours.
c. After cooling down over night (20-22 hours) in room environment as in step a.
d. After vacuum saturation (total time of 1.5 hours) with detailed procedure described below).
e. After air drying over night (20-22 hours) in room environment as in step a.

After each of the strain measurements the samples were weighed. During air drying, the samples were put onto three layers of paper towel to absorb any water present.

This sequence of expansion measurement is more clearly presented in the first five data points on Figure 41. Then the cycles continued to step b again, and so on for a total of 6 cycles. The heating temperature of 110 ± 5 °C was selected in order to ensure that the water adsorbed by the samples could be totally dried out within short time (2 hours).

Vacuum saturation had been found to give effective and fast moisture conditioning. In the vacuum saturation test, the following procedure was carried out: the samples were initially vacuumed without water for 30 minutes, then water was supplied until the samples were fully immersed. The immersed samples were then vacuumed for a further 30 minutes, after that they were left soaking for 30 more minutes.

The results of the first test are shown in Figures 40 and 41. Figure 40 shows that the vacuum saturation caused water adsorption of around 6%. Referring to Figure 41, the first five data points (and the following similar set of steps), were based on strain measurement with sequence as described in step a to e on the first paragraph of this section. Figure 41 shows that after step d-vacuum saturation, then step e-air drying of the first cycle the samples underwent large portion of permanent (irreversible) expansion. From the second and the following cycles, the samples gave almost constant/regular expansion and shrinkage pattern.

In order to clarify the permanent expansion due to vacuum saturation and to evaluate the effect of water adsorption during vacuum saturation test, a second test was carried out. Three samples were tested. The timing of strain measurement was performed as follows:

a. Initially at room temperature (21 ± 1 °C and relative humidity (RH) of 54 ± 4 %).
b. After heating at 110 ± 5 °C for 2 hours.
c. After cooling down for 2 days at room environment as in step 1.
d. After vacuum saturation for (total time of 1.5 hours), then every 24 hours on the following days until the samples stabilized. After that step 2 to 4 were repeated for two more cycles (see Figure 42 for further reference).

The samples used for this second test was the samples from the moisture adsorption (MA) expansion second test as described above. The typical test results are shown in

Figure 43 and 44. In general, Figure 43 shows that the expansion of the samples on their top 't' face were larger than on their base 'b' face. This was because the t face underwent more effective curing.

Figure 44 indicates that face s4 gave slightly higher expansion during air drying (after vacuum saturation). This was because face s4 was positioned on the base of the samples during air drying, therefore the s4 face was always damper than other faces, as the water moves downwards during air drying and less evaporation occurred.

Figure 42 shows more clear average results. Within the first cycle, strain measurement after heating (step 2) the samples expanded to around 900 microstrain. When cooled down (step 3), the samples shrunk almost to their original position. During 2 days cooling down the samples were slightly expanded. This was due to moisture adsorption from the environment as had been experienced. On step 4 the samples were vacuum saturated, where the samples expanded to about 400 microstrain. Then the samples were the air dried at room environment. After vacuum saturation the expansion continued on the two following days to reach about 900 microstrain (on day 4), then started to shrink and stabilized on day 8 to 10. Then the next cycle was continued by heating the samples (step 2), and so on. A very similar strain pattern was obtained.

Due to water exposure (vacuum saturation and expansion during air dryng), the bitublocks expanded between 400-500 microstrain ($10^{-6}$). This moisture expansion is comparable to clay bricks moisture expansion of 280-6000x$10^{-6}$.

Figure 42 revealed that the bitublocks were highly reversible due to thermal exposure. The expansion due to vacuum saturation on the first cycle was found totally irreversible, but largely reversible on the following two cycles. This volume stability behaviour of the bitublocks was unique.

The second test (Figures 42 to 44) confirmed the results obtained earlier in the first test, with additional data on the behaviour of the bitublocks. It was found interesting that during air drying after vacuum saturation, regardless of moisture loss (Figure 40 and 45) the bitublocks experienced expansion then shrunk, and finally stabilized. The maximum water adsorption due to vacuum saturation was found around 6 %.

The following conclusions can be derived from this investigation. The samples produced with a less intense curing regime experienced lower expansion and became stable faster, and vice versa The results suggest that the expansion was caused by moisture adsorption from the environment. Under thermal conditions (at 70°C cycles), the samples with a less intense curing regime, underwent higher expansion, and vice versa. Thermal expansion of the bitublocks was found highly reversible, with coefficient of thermal expansion slightly lower than clay brick and concrete masonry block. The physical stability of the bitublocks was found unique under water exposure. The expansion on first vacuum saturation cycle was irreversible, then largely reversible on the following two cycles. The moisture expansion of the bitublocks was comparable to clay bricks.

**COMPARATIVE EXAMPLE**

Further experiments were carried out to confirm the effect of temperature on the compressive strength of bitublocks and to determine the role of oxidation / volatilization of bitumen on hardening. The results presented below relate to bitublocks, some prepared using methods in accordance with the present invention, and others that do not, but which are very closely related thereto. Consequently, the following results illustrate advantageous features of bitublocks prepared using methods according to the present invention.

The materials used to make the samples included natural silicate sand which was obtained from Tarmac Northern Ltd and which acted as a fine aggregate, PFA and bitumen. Samples made from the fine mix (sand/PFA = 85/15 % by mass; bitumen content = 9.1 %) (See Table 17) were compacted at 8 MPa and 20 MPa (16.5 % and 11.5 % porosity, respectively).

| Bitublock mix type | Crushed glass (10-5mm) | Sand (< 5mm) | IBA (< 5mm) | PFA (< 0.075mm) | 50 pen. bitumen |
|---|---|---|---|---|---|
| Fine mix | | 85 | | 15 | 9.1 |
| Coarse mix | 30 | | 63 | 7 | 10.2 |

**Table 17**

These samples were then cured using the following regimes:

1. Uncured,
2. Cured at 110°C; 120°C; and 130°C for 5 days in the convection ovens at atmospheric pressure,
3. Cured at 110°C; 120°C; and 130°C for 5 days in a PAV at 2.1 MPa.

The average compressive strength results are summarized in Table 18.

| | Compressive strength (MPa) following curing at: | | | |
|---|---|---|---|---|
| Porosity / Pressure | Uncured | 110°C/5days | 120°C/5days | 130°C/5days |
| 16.5% / Atmospheric | 7.5 | 10.9 | 13.5 | 14.6 |
| 16.5% / 2.1MPa | 7.5 | 10.4 | 16.8 | 21.5 |
| 11.5% / Atmospheric | 11.8 | 13.6 | 19.5 | 21.9 |
| 11.5% / 2.1MPa | 11.8 | 12.6 | 19.8 | 28.1 |

**Table 18**

The increase in compressive strength of the samples cured using the different regimes is shown in Figure 46 relative to the control sample.

As can be seen in Table 4 and Figure 46, the compressive strength increased as the temperature increased for both porosities tested. The compressive strength of the samples cured in normal convection ovens was lower than that of the samples cured at 120°C and 130°C under an applied air pressure of 2.1 MPa. The increase in compressive strength of samples with the porosity of 16.5 % cured under pressure in the vessel at 130°C for 5 days was approximately 50 % higher than that of the sample cured in the convection ovens. These results indicate that curing at elevated pressures will enhance hardening, possibly as a result of deeper penetration of oxygen into the specimen pores and the bitumen film. It will be appreciated that autoclave temperatures used in conventional methods for curing standard masonry units under pressure are typically around 300 to 400 °F, but employ pressures of up to around 12 bars, which are therefore significantly higher than the pressures used in the present Example.

The increase in compressive strength of samples with 16.5 % porosity was higher than that of the samples with 11.5 % porosity for both curing regimes (i.e. in the ovens and in the vessel). The results, once again, show that more porous samples obtain greater increases in compressive strength. The influence of curing may therefore be limited by porosity.

The air volume required to oxidize the bitumen film thickness in one kilogram of the fine and coarse Bitublock mixes is presented in Table 19.

**Table 19. Surface area and oxygen requirements for 1 kg of typical Bitublock mixtures**

| Mix Type | Surface area ($m^2$) | Bitumen film thickness (μm) | Factor * ($x10^{-3}$ $m^3/m^2$) | Oxygen absorbed ($10^{-3}$ $m^3$) | Air required** ($10^{-3}$ $m^3$) |
|---|---|---|---|---|---|
| | (1) | (2) | (3) | (4) = (1) x (3) | (5) = (4)/0.21 |
| Fine mix | 10.94 | 9.86 | 3.2 | 35 | 167 |
| Coarse mix | 6.52 | 13.60 | 3.2 | 21 | 99 |

*(*) Beyond 4 μm bitumen film thickness the volume of oxygen absorbed is assumed to remain constant at $3.2 \times 10^{-3}$ $m^3/m^2$ bitumen surface,*
*(**) Air composes of 21% oxygen by volume* (URL2).

The internal volume of the PAV was approximately 7.22 litres. According to the general gas law (URL3), at a pressure of 2.1 MPa in the PAV, the air volumes in the PAV minus the volume of the cured sample are approximately 104; 101; and 98 litres at curing temperatures of 110; 120, and 130°C, respectively. Thus according to Table 19, these air volumes are lower than the volume required for full absorption by the fine mix samples. It is predicted that the results obtained from the PAV curing conditions could therefore have been further enhanced if the trapped air volume in the vessel was greater.

According to Figure 47 the mass loss of the bitumen depends strongly on the curing temperature.

The pure bitumen samples cured up to 100°C resulted in very little mass loss. This also resulted in an insignificant improvement in compressive strength of the Bitublock. At 160°C the mass loss was significantly higher than at 100°C, mainly occurring during the

first 12 hours beyond which there was a definite slowing down. This trend also agrees with the compressive strength curve in Figure 48.

A further experiment was designed to assess the role of oxidation / volatilization of bitumen in hardening and the resultant compressive strength of Bitublocks. Samples made from the same fine mix as described above (sand/PFA = 85/15 % by mass of aggregates; bitumen content = 9.1 %) and were again compacted at 8 MPa (approximately 16.5 % porosity).

These samples were then cured using the following regimes:

1. Uncured;
2. Cured at 160°C for 24, 48, and 72 hours in a conventional convection oven; and
3. Cured at 160°C for 24, 48, and 72 hours in sealed tins.

Compressive strength results obtained from these curing regimes are shown in Figure 49.

The convection oven used in this investigation was a 225 litre capacity oven. Three samples were cured in each batch. Based on the previous calculations, three samples weighing approximately 2.5 kg require an air volume of $420\times10^{-3}$ $m^3$ for complete oxidation of the bitumen binder. With a capacity of 225 litres, combined with efficient air circulation, volatilization and oxidation of bitumen should proceed unimpeded. Meanwhile, with samples cured in the sealed tin vessel having a capacity of only one litre, the volatilization process may occur but oxidation would be severely restricted.

Figure 49 shows a gradual and significant increase (a 500 % increase) from 7.5 MPa for the uncured samples to 45 MPa when samples were cured for 72 hours in the conventional convection oven. In contrast, following similar curing temperatures and durations, samples sealed in the tin vessel achieved only a very small compressive strength increases (approximately 33 %), i.e. from 7.5 MPa to around 10 MPa. These results confirm that the availability of oxygen / air is an important factor in the hardening process of bituminous binders and that the hardening process of a typical penetration grade bitumen seems to be more directly influenced by the oxidation process as opposed to the volatilization behaviour. The results also show that the harsh curing regime adopted for the hardening of bitublock is equivalent to long term hardening

found in asphalt mixes where oxidation is the main hardening process. For the bitumen type in these tests it is confirmed that the volatilization accounts for approximately no more than 7% of the total hardening.

The following conclusions can be drawn from the Comparative Example presented above. The hardening process of a typical penetration grade bitumen (grade 40/60) used in the manufacture of Bitublocks is primarily dependent on its oxidation behaviour. Volatilization behaviour of the penetration grade bitumen does not significantly affect the extent of hardening during curing. The availability of oxygen / air is a very important factor in the hardening process of bituminous binders. Elevated pressures enhance hardening, most likely as a result of the deeper penetration of oxygen into the specimen pores and the bitumen film that can be achieved. Hardening of the bitumen samples in oven aging tests depends strongly upon the curing temperature. At up to 160°C the hardening process occurs slowly but at ≥ 200°C the hardening process occurs rapidly. The bitumen oven aging process occurs mainly during the first 12 hours of heating. However, all samples tested showed that aging does not cease within the first 5 day curing duration selected for this investigation. This was true even when the curing temperature was as high as 240°C. For any bitumen grade and origin, the compressive strength of bitublocks agrees with the mass loss profiles of the bitumen during oven aging. It is suggested that the aging of Bitublocks at temperatures equal to or in excess of 200°C will result in more effective curing than that of lower temperatures. This would therefore enhance the compressive strength (and other properties such as long-term stability) of bitublock. However, it is important to consider that there is a balance between the performance of the new unit and manufacturing energy input. Satisfactory properties (in this case equivalent to current concrete aggregate blocks) can be achieved by curing at the lower temperature of 160°C for 12 hours. Porosity of the compacted bitumen-aggregate mixture is a prime factor controlling bitumen hardening as it controls the access of oxygen / air deeper into the core of bitublock samples and promotes the hardening process.

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## CLAIMS

1. A method for producing a masonry unit, the method comprising forming a mixture containing a bituminous binder and a graded aggregate containing a coarse aggregate fraction, compacting the mixture at a compaction level of up to around 7 MPa, and at least partially curing the compacted mixture.

2. A method according to claim 1, wherein said compaction level is around 0.5 to 5 MPa.

3. A method according to claim 1, wherein said compaction level is around 1 to 4 MPa.

4. A method according to claim 1, wherein said compaction level is around 2 MPa.

5. A method according to any preceding claim, wherein the mixture comprises up to around 10 wt % bituminous binder.

6. A method according to any one of claims 1 to 4, wherein the mixture comprises around 6 wt % bituminous binder.

7. A method according to any preceding claim, wherein the bituminous binder comprises 50 penetration grade bitumen.

8. A method according to any preceding claim, wherein the graded aggregate contains around 20 to 60 wt % of the coarse aggregate fraction.

9. A method according to any one of claims 1 to 7, wherein the graded aggregate contains around 40 wt % of the coarse aggregate fraction.

10. A method according to any preceding claim, wherein said mixture is produced by heating and mixing the graded aggregate and the bituminous binder.

11. A method according to claim 10, wherein at least one of the graded aggregate and the bituminous binder is heated to a temperature of up to around 200 °C.

12. A method according to claim 10 or 11, wherein said at least one of the graded aggregate and bituminous binder is heated for a time period of up to around 5 hours.

13. A method according to any preceding claim, wherein said at least partial curing of the compacted mixture is effected by heating the compacted mixture to a temperature of up to around 250 °C.

14. A method according to any one of claims 1 to 12, wherein said at least partial curing of the compacted mixture is effected by heating the compacted mixture to a temperature of around 80 to 240 °C.

15. A method according to any one of claims 1 to 12, wherein said at least partial curing of the compacted mixture is effected by heating the compacted mixture to a temperature of around 200 °C.

16. A method according to any preceding claim, wherein said at least partial curing of the compacted mixture is effected by heating the compacted mixture for a time period of up to around 72 hours.

17. A method according to any one of claims 1 to 15, wherein said at least partial curing of the compacted mixture is effected by heating the compacted mixture for a time period of around 24 hours.

18. A method for producing a masonry unit, the method comprising forming a mixture containing a graded aggregate and around 6 wt % of a bituminous binder, compacting the mixture, and at least partially curing the compacted mixture.

19. A method according to claim 18, wherein the graded aggregate contains a coarse aggregate fraction.

20. A method according to claim 19, wherein the graded aggregate contains around 40 wt % of the coarse aggregate fraction.

21. A method according to claim 18, 19 or 20, wherein said at least partial curing of the compacted mixture is effected by heating the compacted mixture to a temperature of up to around 250 °C.

22. A method according to claim 18, 19 or 20, wherein said at least partial curing of the compacted mixture is effected by heating the compacted mixture to a temperature of around 200 °C.

23. A method for producing a masonry unit, the method comprising forming a mixture containing a graded aggregate and a bituminous binder, compacting the mixture, and at least partially curing the compacted mixture by heating the compacted mixture to a temperature of around 200 °C.

24. A method according to claim 23, wherein the graded aggregate contains a coarse aggregate fraction.

25. A method according to claim 24, wherein the graded aggregate contains around 40 wt % of the coarse aggregate fraction.

26. A method according to claim 23, 24 or 25, wherein the mixture comprises up to around 10 wt % bituminous binder.

27. A method according to claim 23, 24 or 25, wherein the mixture comprises around 6 wt % bituminous binder.

28. A method of producing a masonry unit substantially as hereinbefore described.

29. A masonry unit produced according to a method defined in any preceding claim.

30. A masonry unit substantially as hereinbefore described.

31. A masonry structure comprising a plurality of masonry units and a mortar, at least one of said masonry units comprising a graded aggregate and a bituminous binder.

32. A masonry structure according to claim 31, wherein the at least one of said masonry units comprises up to around 10 wt % bituminous binder.

33. A masonry structure according to claim 31, wherein the at least one of said masonry units comprises around 1 to 8 wt % bituminous binder.

34. A masonry structure according to claim 31, wherein the at least one of said masonry units comprises around 6 wt % bituminous binder.

35. A masonry structure according to any one of claims 31 to 34, wherein said graded aggregate comprises a coarse aggregate.

36. A masonry structure according to claim 35, wherein the graded aggregate contains around 40 wt % of the coarse aggregate.

37. A masonry structure according to any one of claims 31 to 36, wherein the graded aggregate comprises around 80 to 40 wt % of a fine aggregate.

38. A masonry structure according to any one of claims 31 to 36, wherein the graded aggregate comprises around 50 wt % of a fine aggregate.

39. A masonry structure according to any one of claims 31 to 38, wherein the graded aggregate comprises up to around 10 wt % of a filler material.

40. A masonry structure according to any one of claims 31 to 39, wherein the mortar comprises cement, lime and/or sand.

41. A masonry structure according to any one of claims 31 to 40, wherein the masonry structure exhibits a 60-day creep coefficient of around 2.1.

42. A masonry structure according to any one of claims 31 to 41, wherein the masonry structure exhibits shrinkage over time.

43. A masonry structure substantially as hereinbefore described.

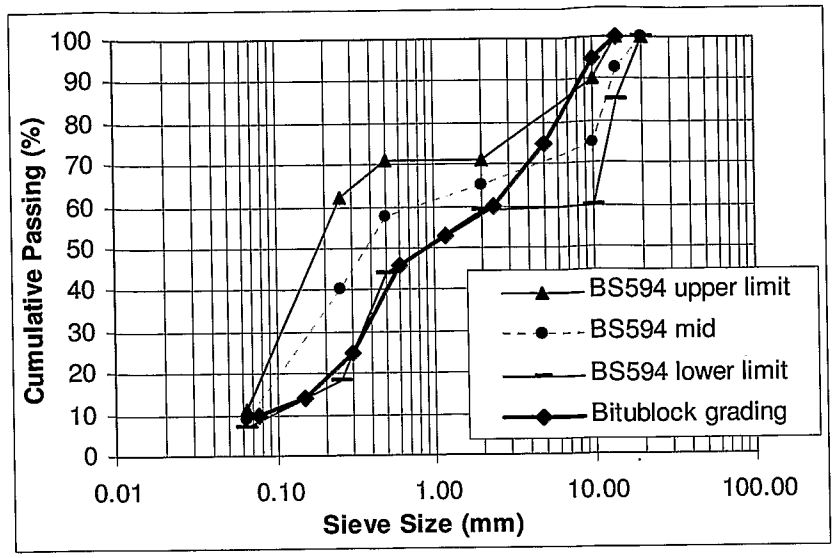



**Figure 1**

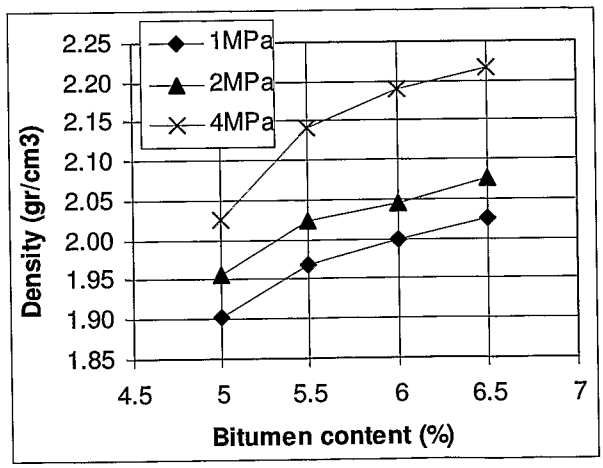



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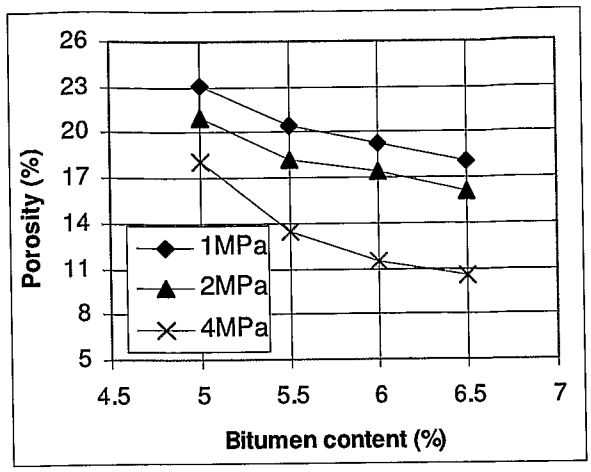



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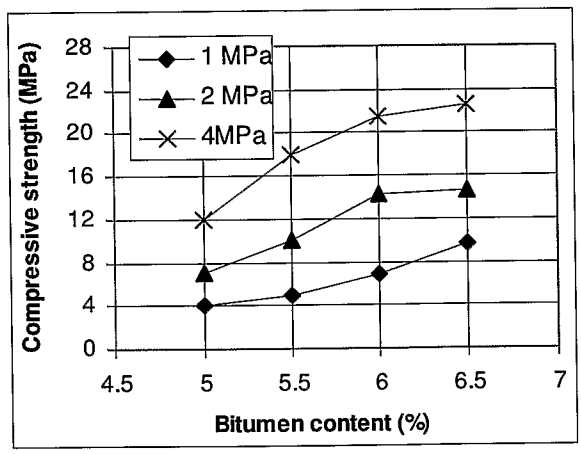



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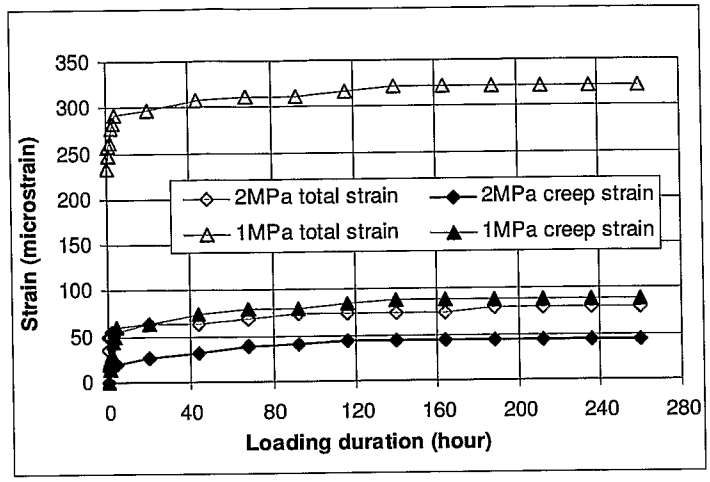



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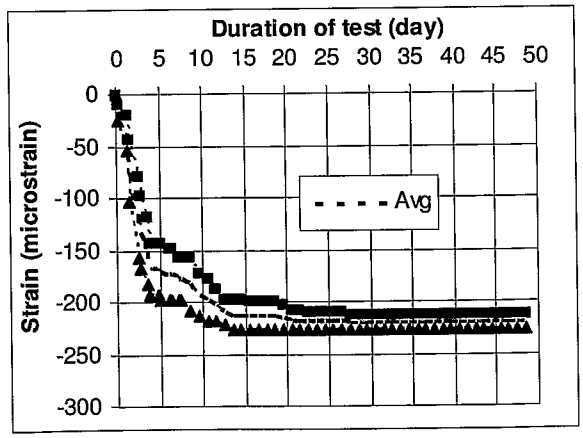



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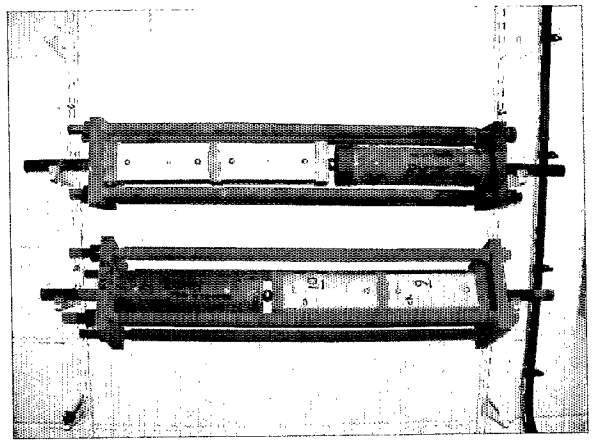


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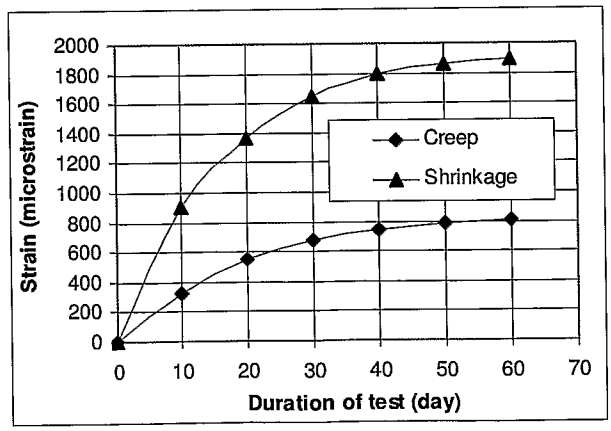



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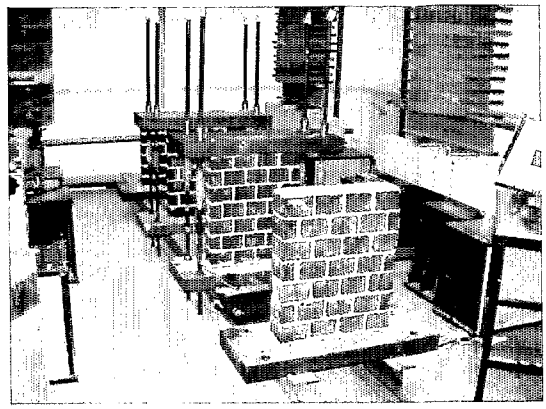


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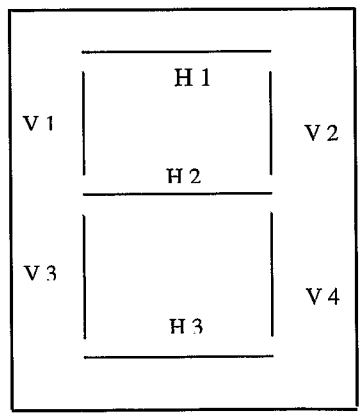



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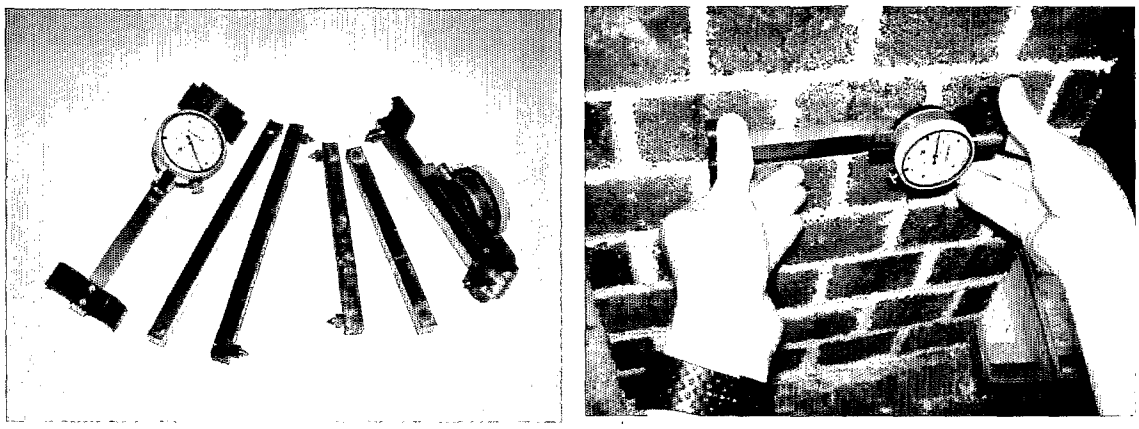


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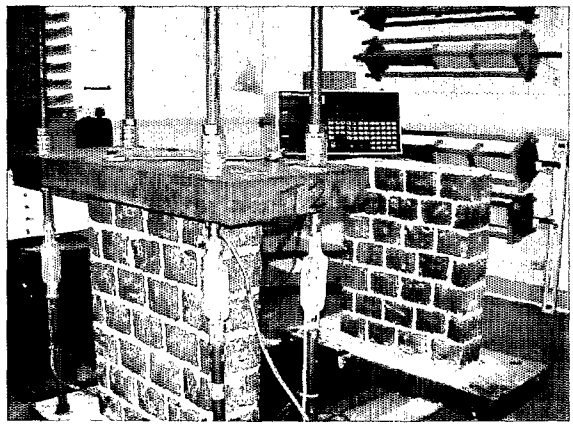


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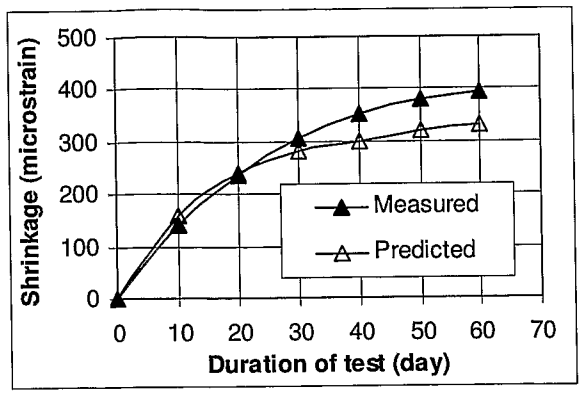



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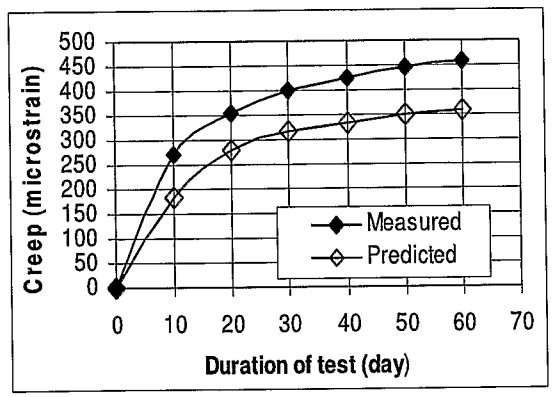



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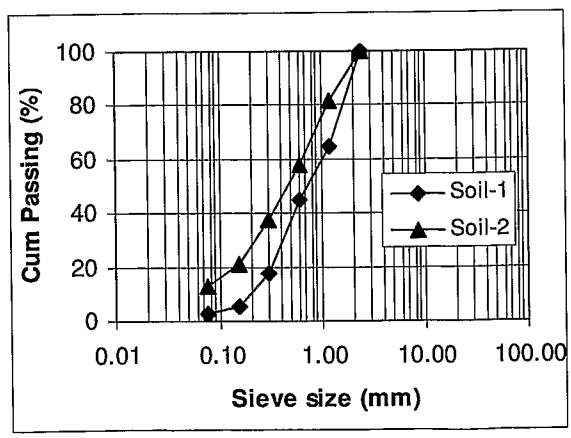



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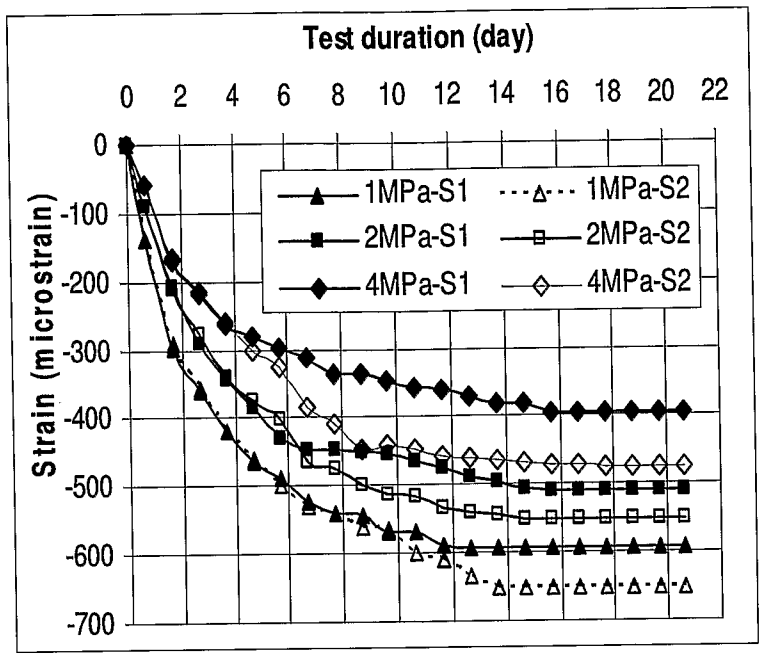



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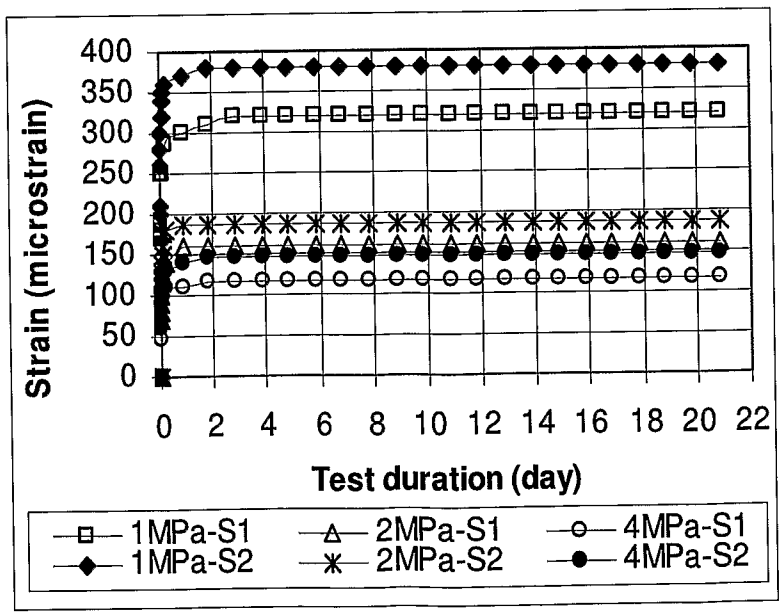



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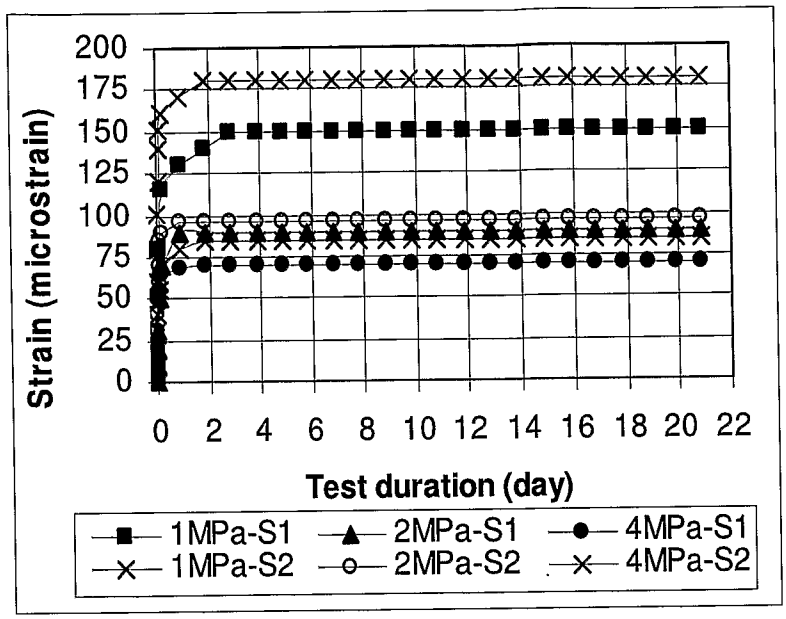



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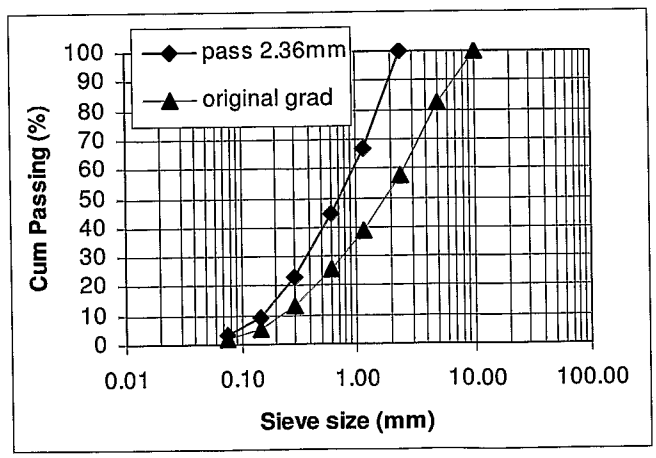



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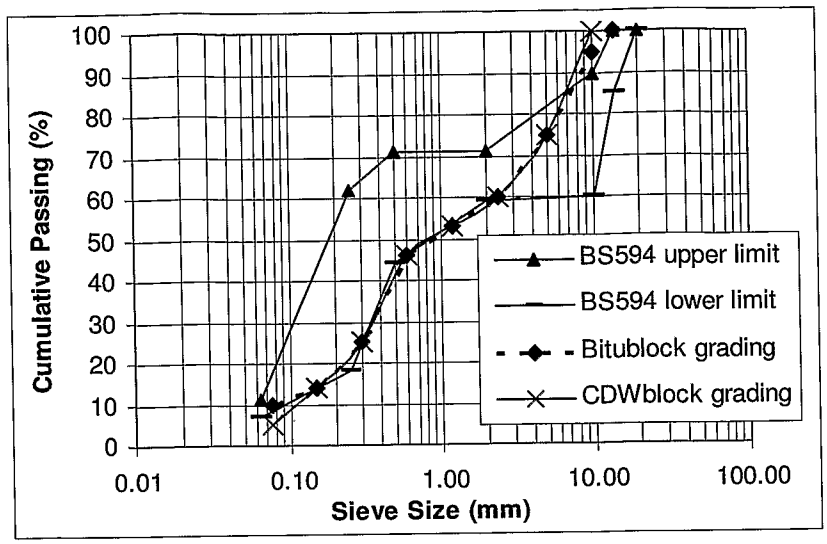



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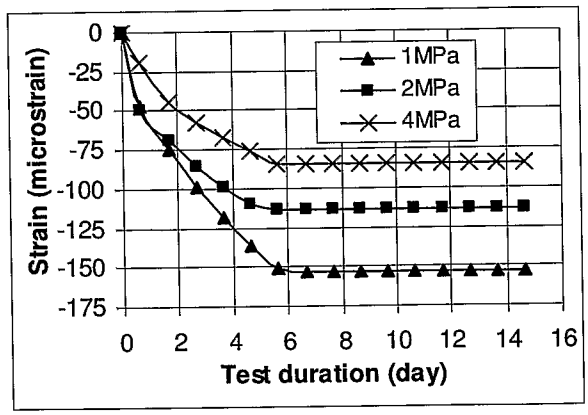



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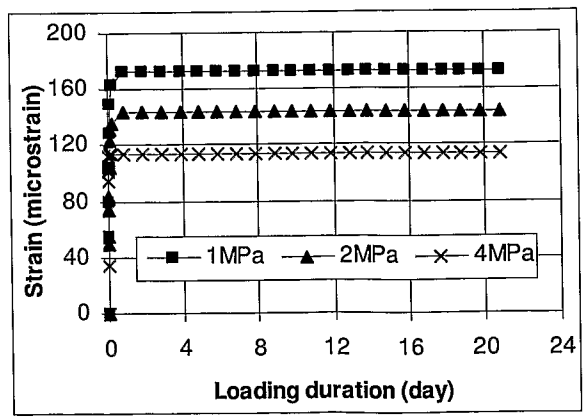



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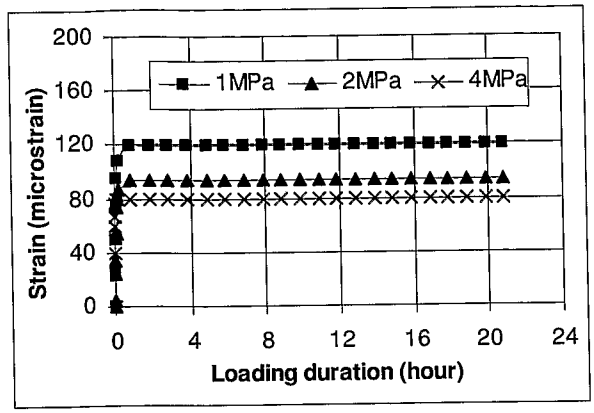



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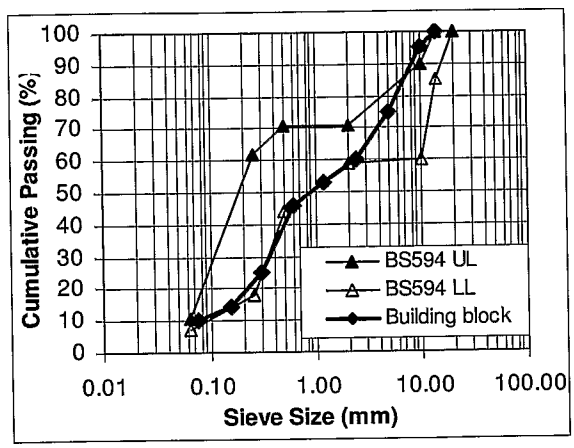



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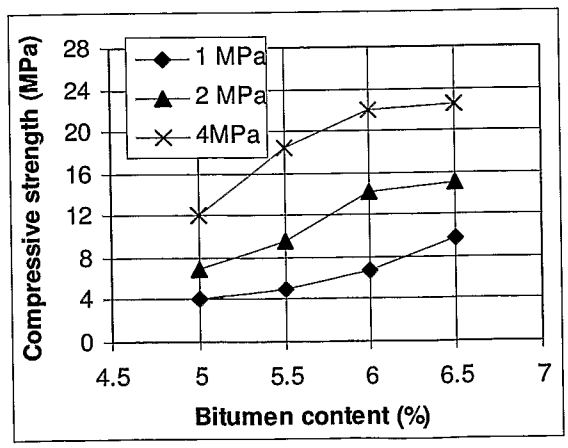



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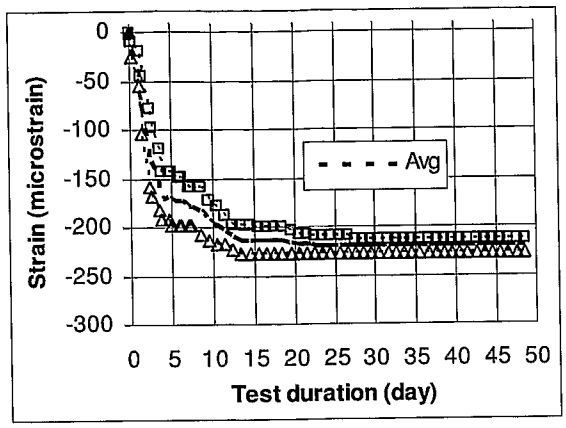



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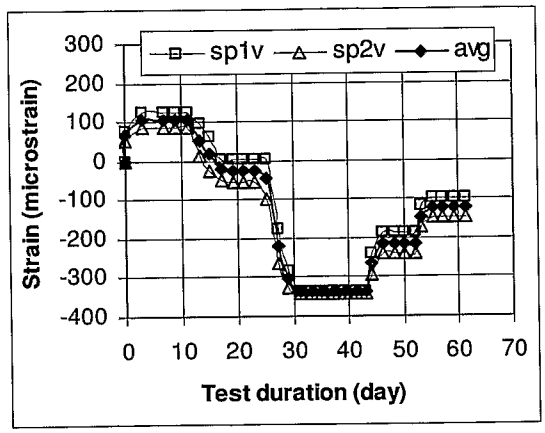



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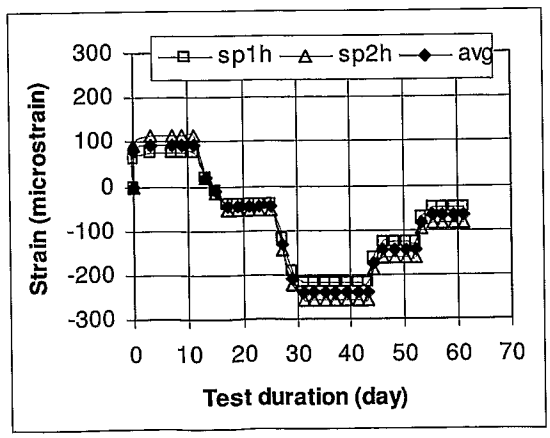



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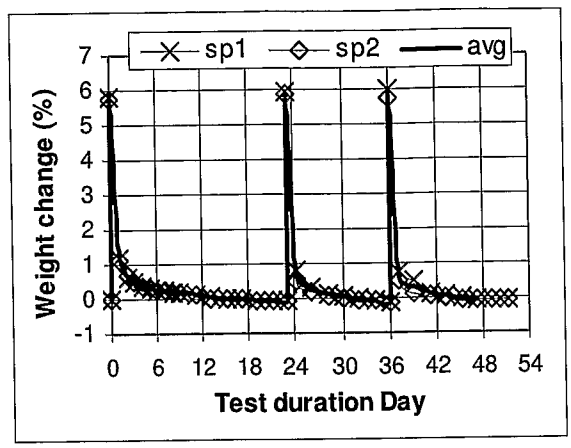



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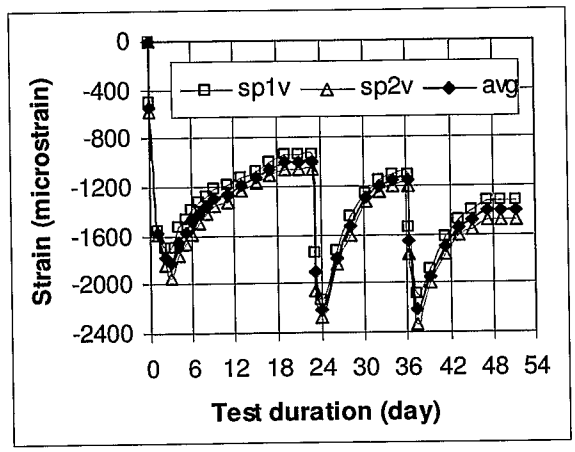



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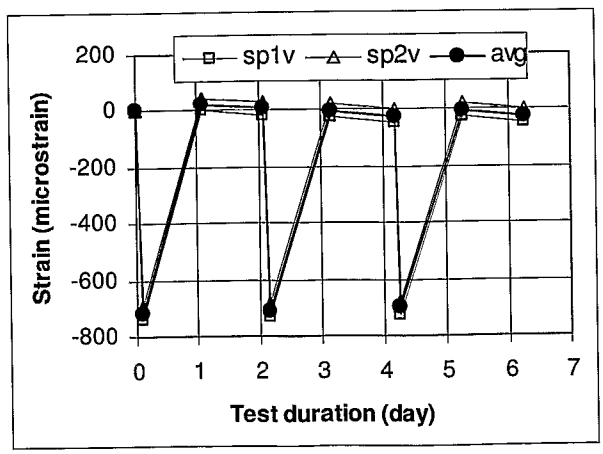



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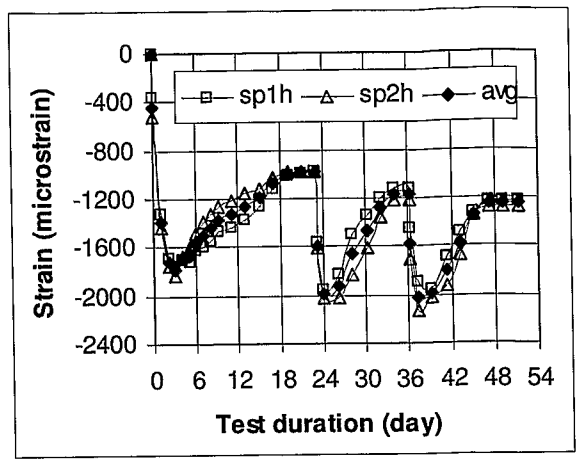



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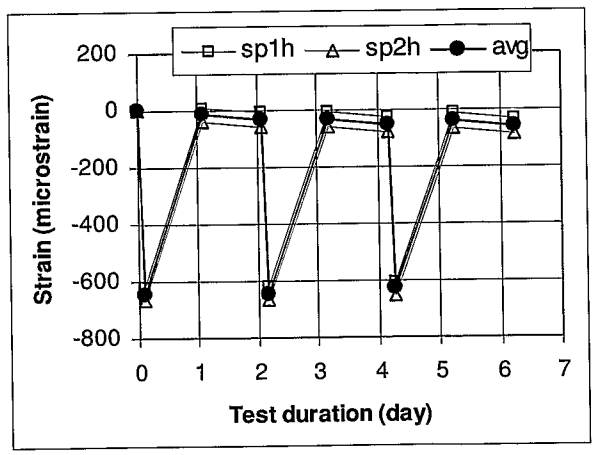



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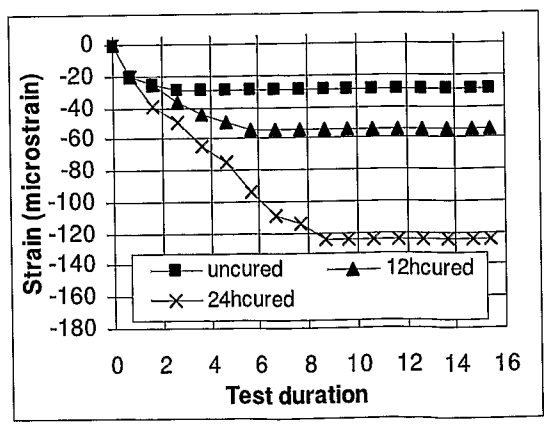



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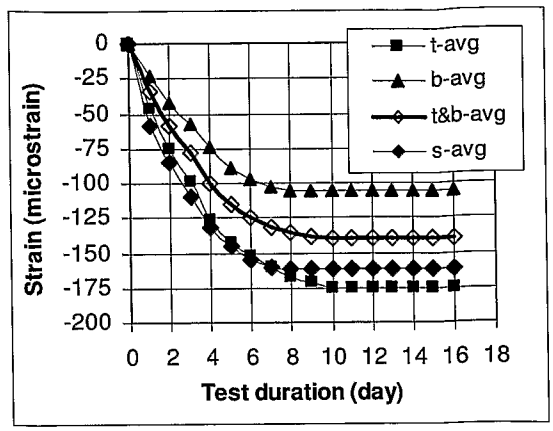



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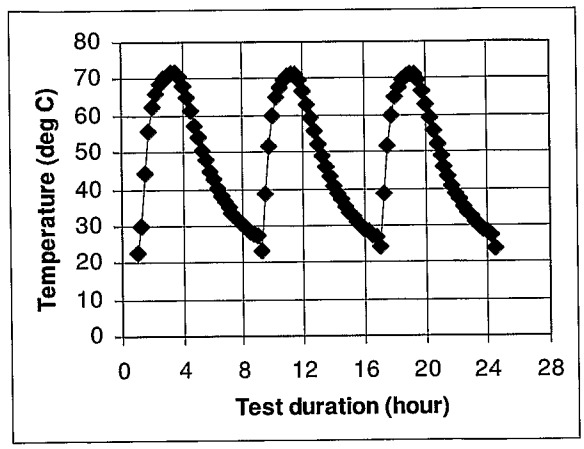



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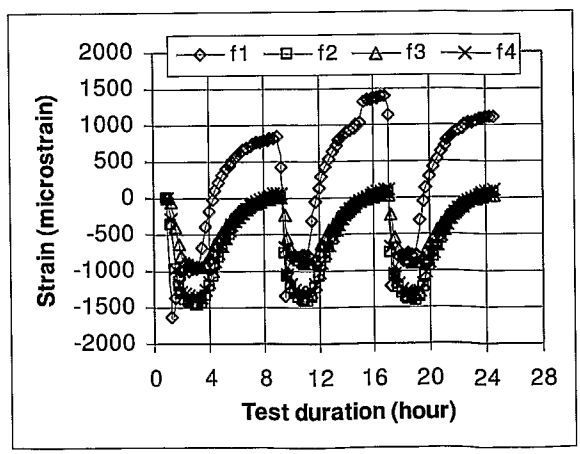



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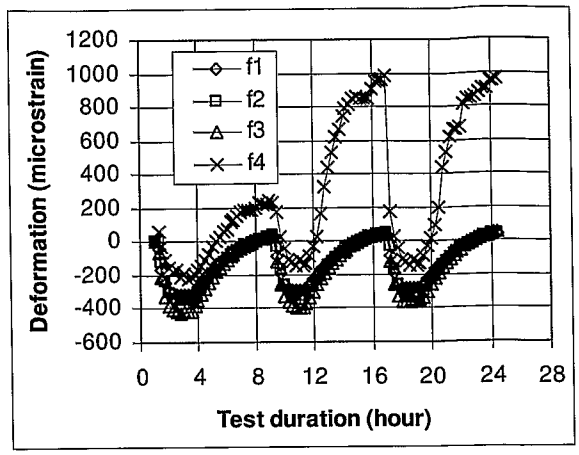



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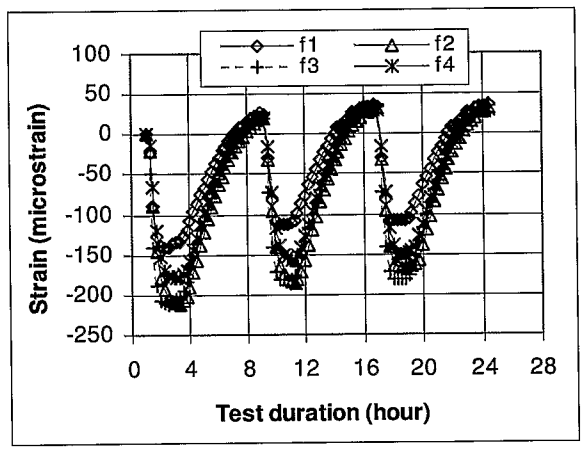



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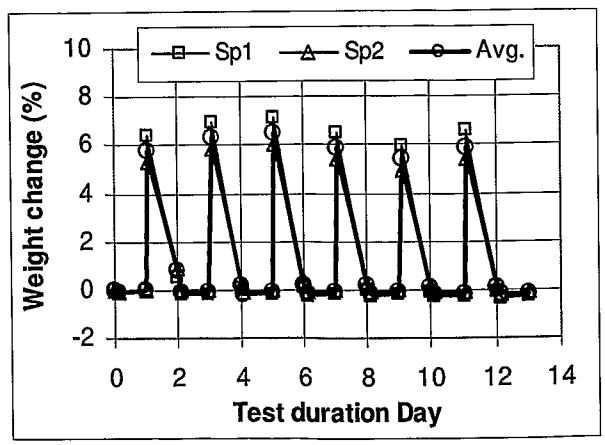



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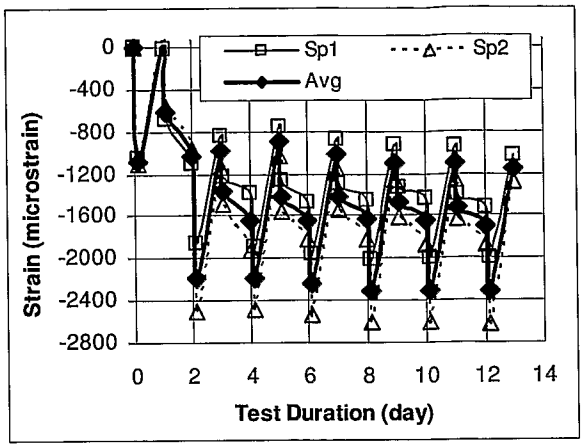



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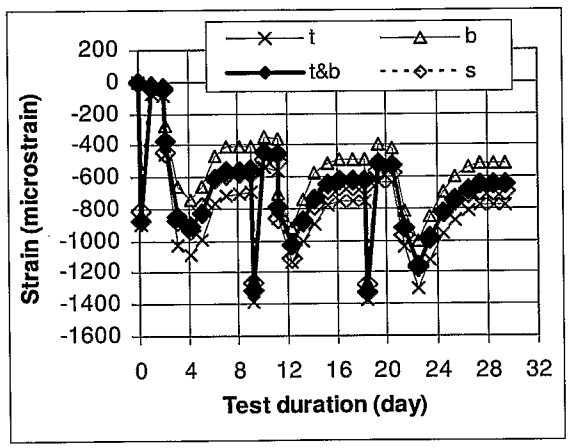



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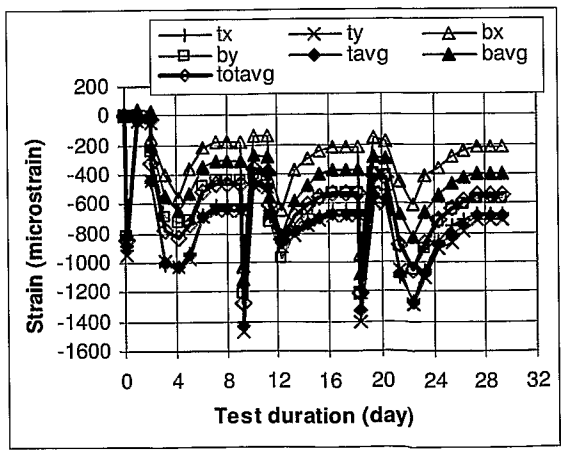



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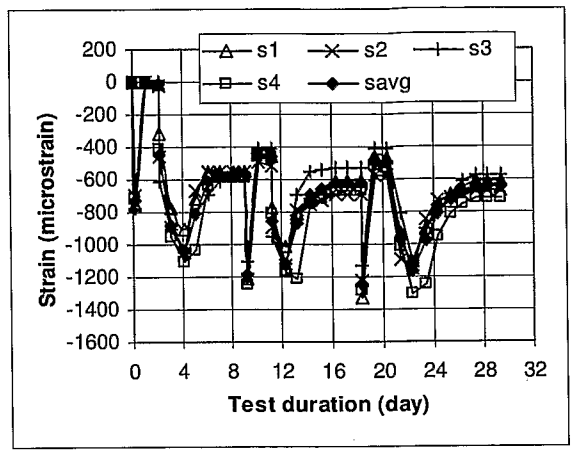



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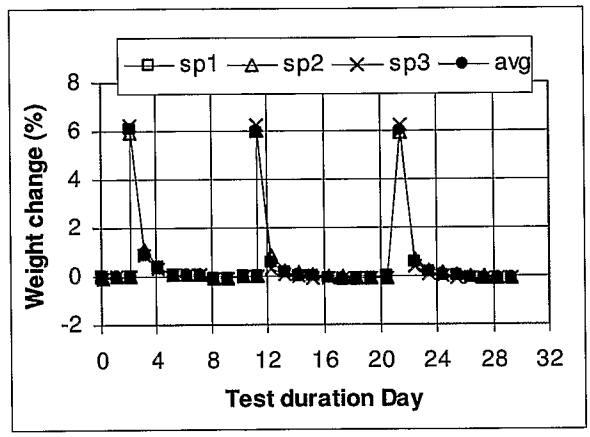



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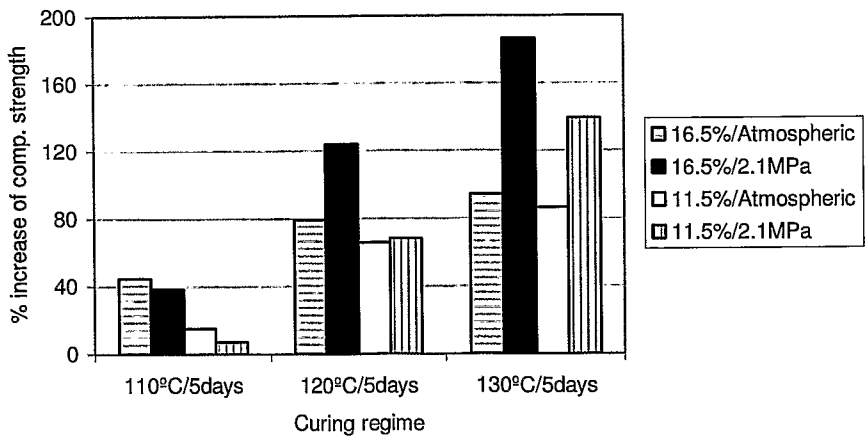



Figure 46

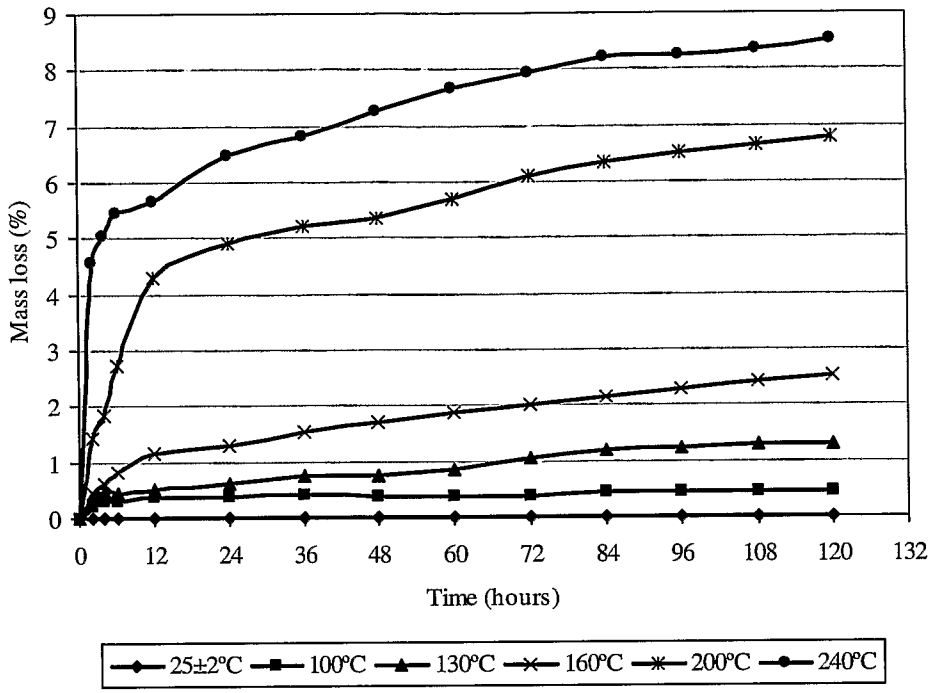



**Figure 47**

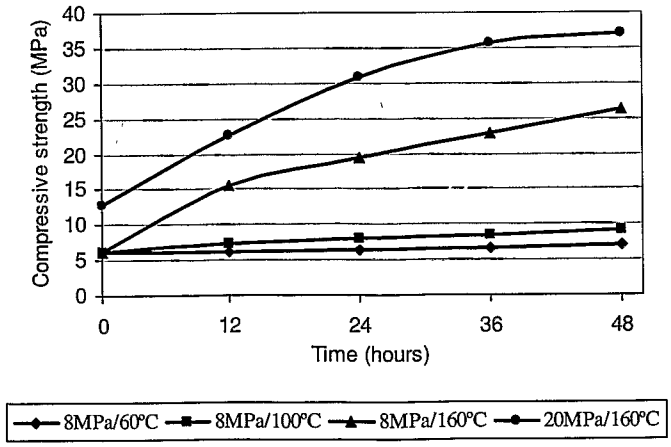



**Figure 48**

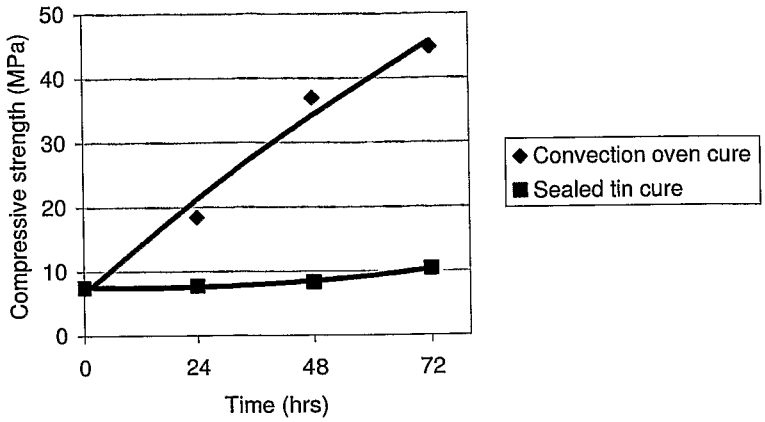

50
40
30
20
10
0
Compressive strength (MPa)
0
24
48
72
Time (hrs)
Convection oven cure
Sealed tin cure

**Figure 49**

# INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2009/000347

**A. CLASSIFICATION OF SUBJECT MATTER**
INV. C04B26/26

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)
B09B C04B E01C C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| X | FORTH, J.P., ZOOROB, S.E. & DONG VAN DAO: "The Development of a Masonry Unit Composed Entirely of Recycled and Waste Aggregates" INTERNATIONAL RILEM CONFERENCE ON THE USE OF RECYCLED MATERIALS IN BUILDINGS AND STRUCTURES, November 2004 (2004-11), pages 341-350, XP008106555 chapter 2 page 342 - page 344 chapter 3.4 page 345 figures 2,3; tables 2,3,4a,4b,5a,5b | 1-5,7,8, 10,12,14 |

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

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"&" document member of the same patent family

| Date of the actual completion of the international search | Date of mailing of the international search report |
|---|---|
| 25 May 2009 | 18/06/2009 |

| Name and mailing address of the ISA/ | Authorized officer |
|---|---|
| European Patent Office, P.B. 5818 Patentlaan 2<br>NL - 2280 HV Rijswijk<br>Tel. (+31-70) 340-2040,<br>Fax: (+31-70) 340-3016 | Büscher, Olaf |

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# INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2009/000347

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International application No
PCT/GB2009/000347

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International application No
PCT/GB2009/000347

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