JOURNAL OF MATERIALS SCIENCE: MATERIALS IN MEDICINE **15** (2004) 1065–1071

Mechanical characterisation of a bone defect model filled with ceramic cements

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Ceramic bone substitute materials are often used to fill defects in comminuted articular fractures. In an *in vivo* study [1], calcium phosphate cements have been injected into highly loaded slot defects in the proximal tibial metaphysis. During healing, cracks were formed mostly in the proximal anterior aspect of the implanted cement and wedge-like gaps formed between the tibial plateau and the cement. Mechanical *ex vivo* tests were done to investigate the mechanical competence of the bone cement in such a defect situation.

Entirely filled defects were loaded with up to 4.5 kN until they failed. Cyclic loading of the proximal tibiae caused micro fragmentation of the cement after 1000 cycles at 1.5–2.0 kN load. This aspect was comparable to cement fragmentation observed *in vivo*. Large defects in highly loaded areas should therefore additionally be stabilised with metallic implants. The ceramic cement can only be used as a filler material, which can be replaced by new bone upon resorption.

Introduction

Ceramic bone substitute materials are often used for bridging large defects after trauma, tumour resection or in the treatment of pseudarthrosis [2–6]. Until the late 1980s, the material was mainly implanted as blocks or granules. Shaping of the brittle ceramics is a problem and the debris often block the pore structure preventing new bone to grow into the material. Injectable materials came to the market in the mid-1980s. Different calcium phosphate powders are mixed with an aqueous solution to form a paste that hardens *in situ* to a solid material [3, 7]. Filling of cavities in a fracture site is much more convenient with these materials.

Most ceramic bone cements set to a hydroxyapatite (HA) as a final product. HA is the mineral that most resembles the crystalline phase of bone, therefore, it is considered – and proven – to be a highly compatible material [7, 8]. Hydroxyapatite has a very slow resorption rate *in vivo*. The cements stay in the body for more than one year [9, 10]. Fracture healing, however, is much faster in most of the cases [11]. A biphasic cement has been developed by the group around Lemaître at the EPF in Lausanne [12–14]. This material consists of a brushite (DCPD) matrix filled with β-TCP granules. As compared to HA, β-TCP has about an eight times and DCPD even a 15 times higher solubility. This leads to *in vivo* resorption times between six and 18 months.

In an *in vivo* study, the performance and resorption patterns of two different ceramic bone cements (biphasic DCPD–β-TCP and monophasic HA) were investigated in different defects [1]. In a drill hole in the femoral condyles, both cements showed the expected results with different resorption patterns: the monophasic HA cement showed minor superficial resorption with good integration in the surrounding bone; the biphasic cement showed a corresponding resorption pattern with faster degradation of the matrix as compared to the granules, which were then surrounded by newly formed bone. In a highly loaded proximal tibial slot defect, cracks started to form in the anterior part, which were immediately filled with new bone. However, the mechanical competence of this situation and the origin of the cracks – mechanically or biologically induced – are not known. A mechanical *ex vivo* test should clarify this situation.

Aim of the study

In the context of the *in vivo* study, the following questions should be answered:

1. How big are the loads, which can be taken by a tibial slot defect [1] that is left empty or is filled with a ceramic bone cement?
2. What is the effect of an incomplete filling of the defect on the load bearing capacity of the defect?
3. How does cyclic loading change the cement's load bearing capacity?

Materials and methods

Bones for testing

Sixteen cadaver tibiae of Swiss Alpine Mountain Sheep (12 left, 4 right) were used for this study. They have been taken from untreated legs of other studies carried out

TABLE I Group assignments of the tested bones. Bone 07 was used to evaluate the test and measurement procedure

	Static test	Dynamic test (with gap)
Norian®	bone 05 bone 06 bone 08	bone 04 bone 09 bone 10
Gypsum	bone 11 bone 12 bone 16	bone 13 bone 14 bone 15
Empty defect	bone 01 bone 02 bone 03	

earlier. The average age of the bones was 60 months (31–112 mo). They were wrapped in saline soaked gauze and frozen at − 22 °C.

As implant materials, two cements have been used:

- Norian®SRS®, Norian Corp., Cupertino CA, USA (Lot-Nr. 2000-02/019); carbonated apatite
- Calcium sulphate, Fluka Chemika, Buchs, Switzerland (Lot-Nr. 398533/1-14699)

The distal part of the bones was first cut off (14.5 cm distal to the landmarks described in Gisep *et al.* [1]) and embedded in Beracryl® (cylinder of 48 × 60 mm). At the proximal end, the slot-shaped defects were created between the attachment of the patellar ligament on the tibial tuberosity and the tibial plateau. Attachment sites of medial and lateral tendons were used as landmarks. The slot defect had a height of 6 mm and was penetrating about 50% of the lateral projection. The posterior cortex was left intact.

The bones were assigned to the following groups:

About 15 bones were used for the testing, three in each group. Bone 07 was used to evaluate the test and measurement procedure. The slot defects were filled according to Table I.

Gypsum was mixed in a 2 : 1 powder : liquid ratio (30 g powder, 15 ml dist. H_2O). It was mixed for 30 s in a vacuum-mixing machine (Degussa R11 mixer/vibrator), filled into a 5 ml syringe and injected into the defect through a 14 ga needle. After 30 min of setting, the bones were again wrapped in saline soaked gauze and stored at − 22 °C. For the dynamic tests, a 1 mm thick metal sheet was placed on the posterior border of the defect and the gypsum was injected like in the other defects. After setting of the material, the metal sheet was removed leaving a 1 mm gap between the cement and the tibial plateau.

Norian®SRS pouches were mixed in a standard pneumatic mixing machine (MXR-PNE01-UNV) provided by Norian Corp. The material was also filled into a 5 ml syringe and injected into the defect using 14 ga needles. For setting, the bones were wrapped in saline soaked gauze and stored for 24 h at 37 °C in an incubator. The gap models were produced in the same way as with the gypsum.

For the proximal embedding, two maxillofacial screws (stainless steel, 2 × 10 mm) were placed in the anterior part of the tibial plateau. These screws were then embedded in Beracryl®. On the top surface of the Beracryl® block, a metal plate ($15 \times 15 \times 3\,mm^3$) was placed to allow for a force introduction with a centre punch. Compression forces were introduced on the posterior edge of the slot defect in the middle of the medio-lateral aspect of the tibia (Fig. 1).

Mechanical testing and evaluation system

For mechanical testing, an Instron 4302 compression machine was used. For all tests, the bone was placed upright in the Instron, on a *xy*-table with free rotation around the *z*-axis allowed. The static tests of the entirely filled and the empty defects were carried out with a crosshead speed of 1 mm/min. The load was measured by a 10 kN loadcell and calculated on a Windows computer system (Instron Series IX Version 8.06.00 software). Failure of the bone or bone-implant system was taken as a stop criterion.

The dynamic tests were carried out on the samples with a 1 mm gap between the plateau and the cement. In a load-controlled manner, 1000 cycles at loads between 1500 and 2000 N were applied to the system. Crosshead speed was 15 mm/min. The amount of load was chosen in such a way that the upper part of bone (tibial plateau) touched the cement surface. Depending on the size of the bone, the load had to be adjusted.

To detect the real deformations in the specimens and the defects, an optical measuring system was integrated into the test setup. Two video cameras (Kappa, CF 15DSP RGB) were placed next to the Instron machine to observe the gap from anterior (camera A) and lateral (camera B). Points were marked on the bone surface with a felt pen. Digital images of the defect were taken in 1 or 2 s. intervals (TimeLapse, Carl Zeiss GmbH, Germany). The relative movement of these two points was then evaluated using AxioVision® Version 3.1 (Carl Zeiss GmbH, Germany). Calibration was done with a micro-scale. This optical system also allowed for an investigation of the failure mechanism and location. The theoretical pixel resolution of the system was calculated to be between 0.015 and 0.020 mm. Measurements however were not carried out on a pixel level but with an apparent accuracy of 0.10 mm.

To analyse the failure mechanisms of the tibial plateaus, the Beracryl blocks were carefully removed from the tibial plateaus and the bones were investigated macroscopically. Additional to this, the images taken with the video cameras were analysed, especially the ones taken from lateral, to identify where the failure occurred.

Results

Static tests, Instron data

The unfilled tibial defects were loaded up to an average of 1.2 kN (1.1–1.5 kN). There was no brittle kind of failure but a creeping. Bone 01 and 02 showed a very similar force-displacement curve. The slope of the curve – indicating the elasticity of the plateau – was again very similar in bone 03. However, the maximum load was at 1.5 kN. Fig. 2 shows the single curves.

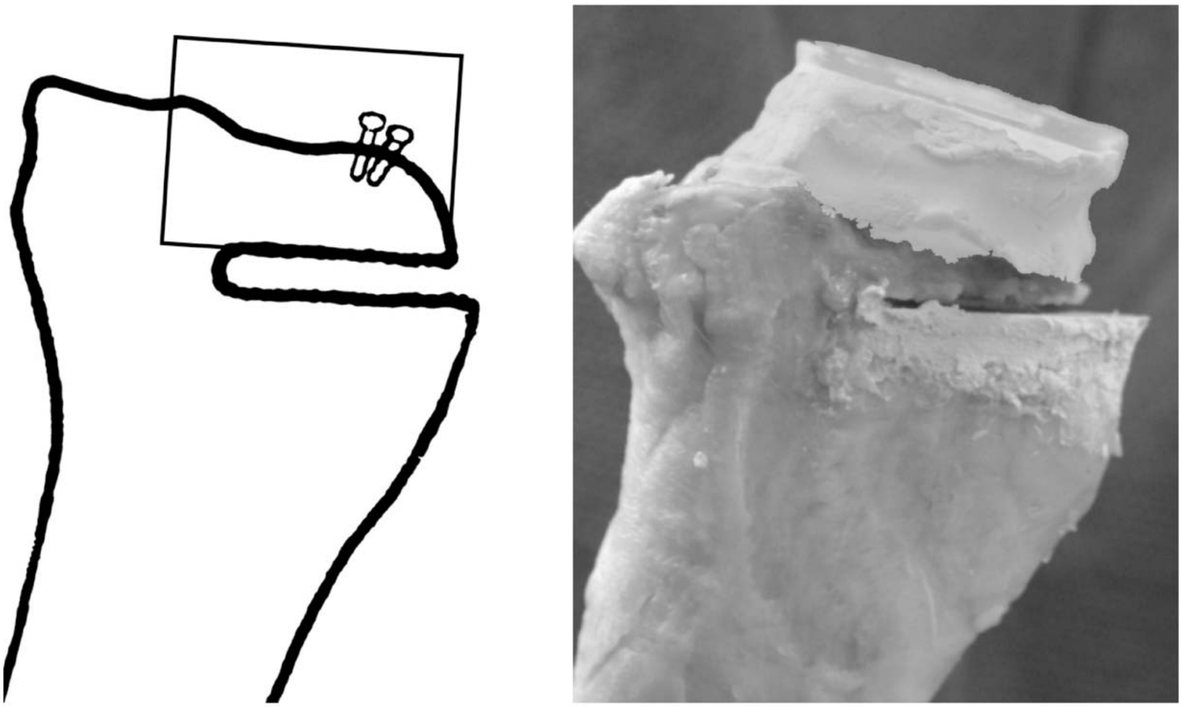

Figure 1 Schematic 2-D drawing of the slot defect in the proximal tibia. On the anterior aspect, two 2.0 mm stainless steel screws were placed to be able to fix the Beracryl-block to the tibial plateau (right). The defect was then filled with the ceramic bone substitute (Norian®SRS® for dynamic testing in this case).

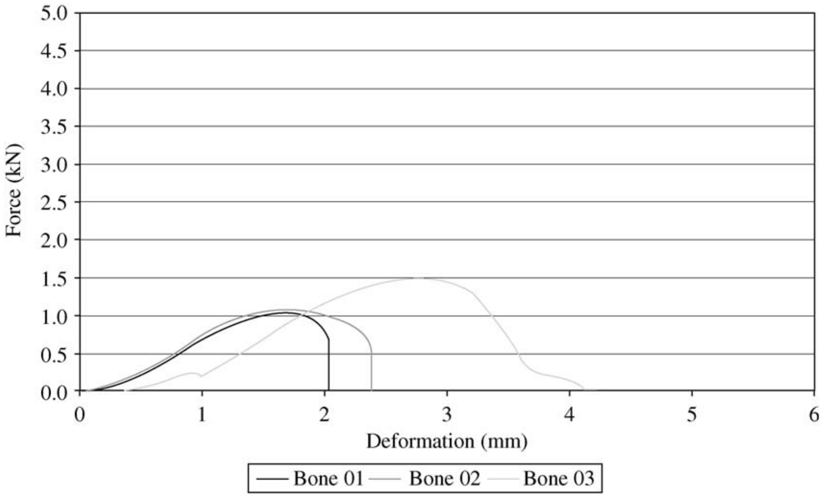

Figure 2 Force – Deformation diagrams of the three tested bones with empty defects. The maximum loads were between 1.1 and 1.5 kN with an average value of 1.2 kN. The stiffness of the single curves is similar.

The defects, which were filled with either Norian SRS or calcium sulphate, were also tested in compression. Norian SRS filling led to maximum forces between 3.1 and 4.1 kN, the average load was 3.7 kN, the same as for the gypsum-filled samples. These values however scattered a little more between 2.9 and 4.5 kN. The stiffness of these six samples again was very similar to each other (Figs. 3 and 4).

Static tests, optical analysis

The optical measurements with the video system revealed differences to the deformations measured by the Instron machine. In the empty defects, the deformations calculated by Instron and optical system were quite similar. However, the deformations given by the Instron were always slightly higher than the ones from the optical system.

In the filled defects, this difference was a lot more obvious. As can be seen in Fig. 4, the deformation calculated by the Instron was about 3–4 mm until the maximum load was reached. The optical system however revealed no deformations up to the maximum load. An example of this is given in Fig. 5, where the Instron and the real deformations in the defect are displayed for bone 12.

Dynamic tests

The dynamic tests – carried out on the defects with a gap on the proximal side – showed, that after 1000 cycles at 1.5–2.0 kN only, cracks had formed mainly in the anterior

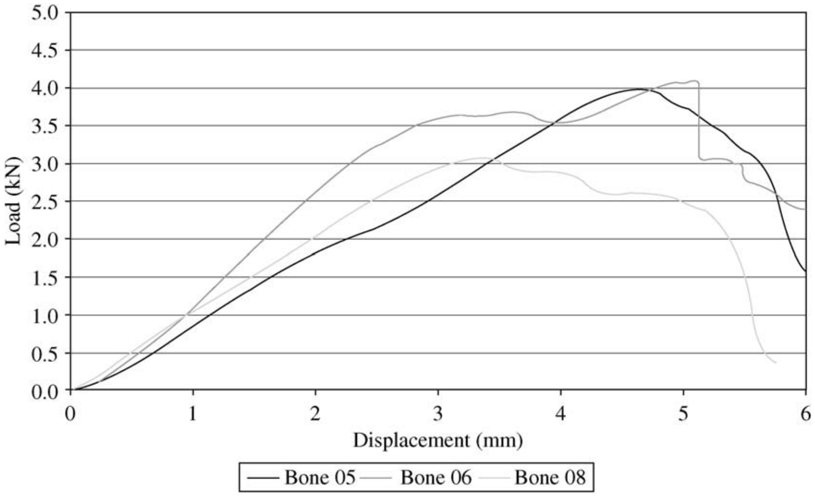

Figure 3 Compression testing of Norian SRS filled defects. The average load was 3.7 kN with a range of 3.1–4.1 kN. The stiffness was similar for all three samples.

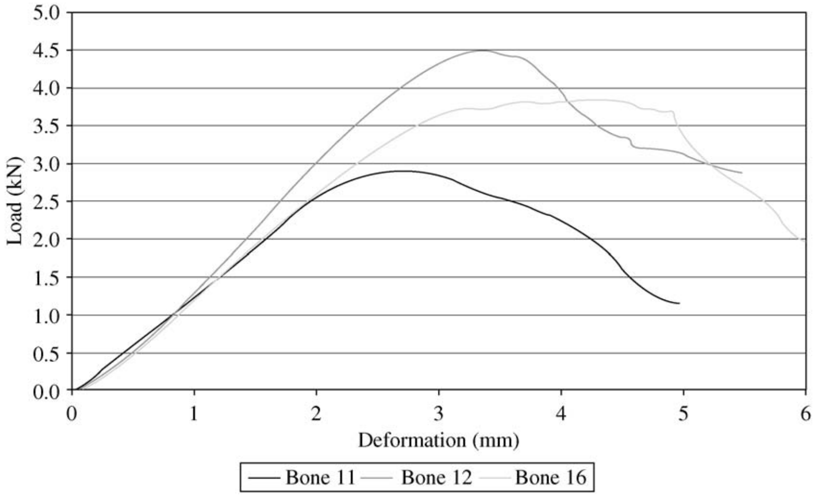

Figure 4 Calcium sulphate filled defects in the compression test: average value of 3.7 kN, range from 2.9 to 4.5 kN. Note the regular slope of all three curves.

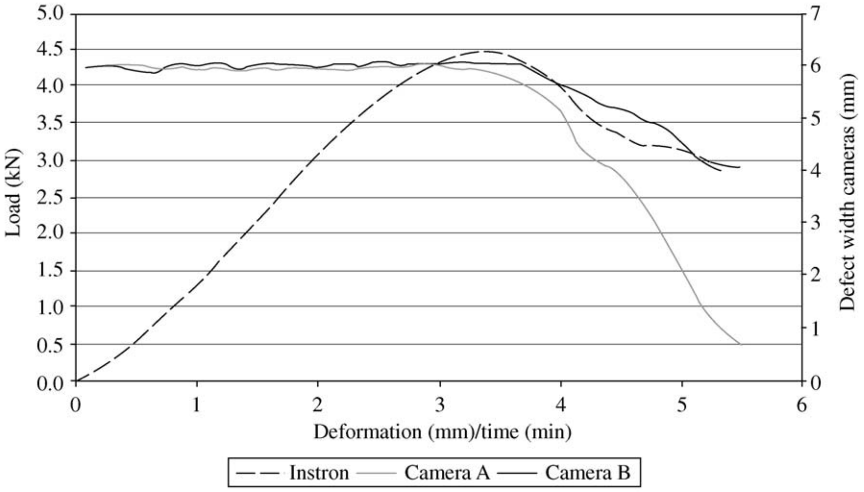

Figure 5 Measurement of the real deformation in the defect by an optical system revealed the stability of the construct. Until a load of 4.5 kN, no deformation occurred in the defect. The Instron however showed a displacement of about 3.5 mm, which has to be attributed to deformation in the supporting construct.

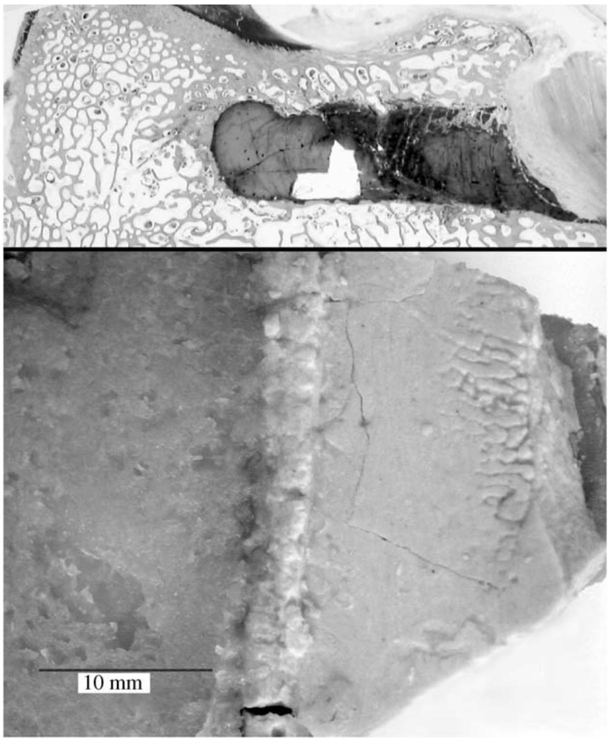

Figure 6 The cracks in the *ex vivo* model (bottom) looked very similar to the ones observed in the histologic section out of the *in vivo* study (top). More cracks and cement particles are formed in the anterior, highly loaded part of the defect whereas in the posterior aspect, the cement still is almost intact. The top part of the picture is a sagittal section through the defect; the bottom part is the view on the cement in a transversal plane.

part of the cement. No difference was seen between the two implant materials Norian SRS and gypsum. The cracks started to form after about 800 cycles in all cases. They first formed in the anterior part, grew bigger and more towards the posterior part. After testing, the tibial plateaus were removed and the same crack and fracture pattern was seen on the implant material as in the *in vivo* study done by Gisep *et al.* [1] (Fig. 6).

Failure mechanisms in the samples

The failure mechanisms were mainly investigated in the samples with empty defects or in the dynamically tested samples with a gap between the tibial plateau and the cement. All but one of the investigated samples did not fail in the plane where the two screws were placed but about 5 mm further posterior. One sample failed in the plane of the inserted screws. On the lateral pictures taken by video camera B, this mechanism was also shown by an angle in the upper surface of the defect (Fig. 7). The tibial plateau did not behave like a homogeneous cantilever beam.

Discussion

Static tests, Instron data

The static tests of the unfilled tibial slot defects revealed the instability applied to the system by creating the defect. A load of 1.21 ± 0.24 kN was enough to destroy the tibial plateau. This load corresponds to about two times body weight of the sheep. This load occurs during normal gait of a sheep on flat ground [15]. However, during running, jumping or walking on uneven ground, it might be exceeded by far.

In the static tests, all defects filled with either Norian SRS or gypsum showed the same behaviour and similar values. Norian SRS however has a compression strength of about 50 MPa [7], whereas calcium sulphate breaks at about 20 MPa [16]. The reason for the similarity in support to the tibial plateau in this study could be the low temperature of the bone, when the material was injected. Norian SRS should set at 37 °C to reach its ultimate strength. During injection of the material into the defect, the bone surface could have had a temperature of below 0 °C due to the storage in the freezer. Time between the moment at which the bone was taken out of the freezer and injection of the cement was kept at less than 5 min to avoid tissue deterioration. This might have disturbed the setting process to such an extent, that the material reached a final strength in the area of the calcium sulphate. The failure strengths of 3.74 ± 0.62 kN were about three times higher than without implant material. This corresponds to about five to six times body weight of the sheep, which is an adequate value for normal activity but could still be exceeded during running or walking on an uneven ground.

Static test, optical analysis

The differences between the deformations measured by the Instron and the real deformations in the defect measured by the optical system were obvious. The empty defects showed quite similar behaviour with the two methods. Deformations were always in the same range, not deviating more than 1 mm from each other. This indicates that the cantilever was the weakest part in the entire system. The filled defects however showed in the optical system no deformations up to the maximum load. This means that the filled defect was stable until then. The deformations measured by the Instron therefore had to occur in the embedding and the fixation system of the Instron itself. The proximal embedding was certainly the weakest point in the system. It was done upside-down, which means that the embedding material was poured

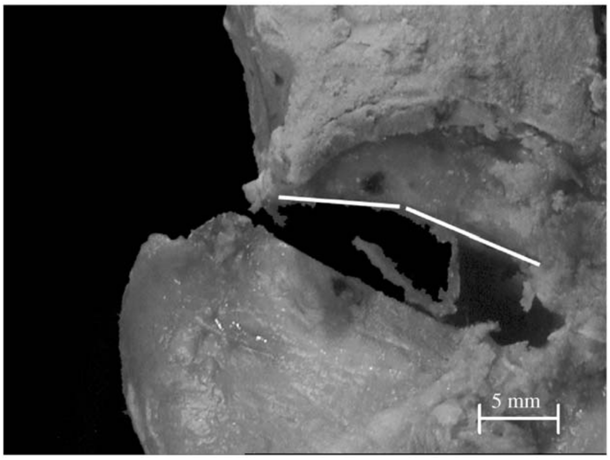

Figure 7 The tibial plateau is not a homogeneous cantilever beam. The two white lines on the tibial plateau indicate the angle in the plateau, located about 5 mm posterior to the insertion plane of the two screws.

into the template and the bone with the two screws pushed into it from above. The form-fit between tibial plateau and embedding might not have been perfect. This connection therefore had to settle first, before the load was really transferred to the tibial plateau. Additionally, the attachments of cruciate and patellar ligaments could not be perfectly removed. These and the articular cartilage could also have acted as soft links between the embedding and the defects. This would explain the differences in the initial phase of the compression test.

In the later stage of the test, the proximal embedding started to tilt towards anterior. This led to a growing difference between the Instron and the real deformation in the defect. However, the different curves were all very similar, which facilitated the interpretation of the curves.

Dynamic tests

The dynamic test revealed a weakness of the system bone-implant at loads around 1500–2000 N. This is about twice the body weight of the sheep. However, these loads led to failure of the implant material in presence of a gap on the posterior part of the defect. In an *in vivo* situation, incomplete defect filling or an initially disturbed balance of cement resorption and bone remodelling could cause this gap. The cement showed cracks, mostly in the anterior part, after only 1000 cycles. The constant loading seems to highly challenge the implant material. The gap on the proximal side of the defect weakens the system further. A stable situation after surgery seems to be crucial for highly loaded defects filled with ceramic bone cements.

Failure mechanisms in the samples

The plane lying about 5 mm behind the two screws placed in the tibial plateau seems to be the weakest in the whole plateau. This could be caused by two reasons. Contact radiographs taken of the bones revealed a discontinuity in the bone architecture exactly at this weakest point. In the tibial plateau, most of the bone trabeculae are oriented horizontally in a sagittal plane. This orientation changes to a more vertical one right at the spot of failure in the samples (Fig. 8).

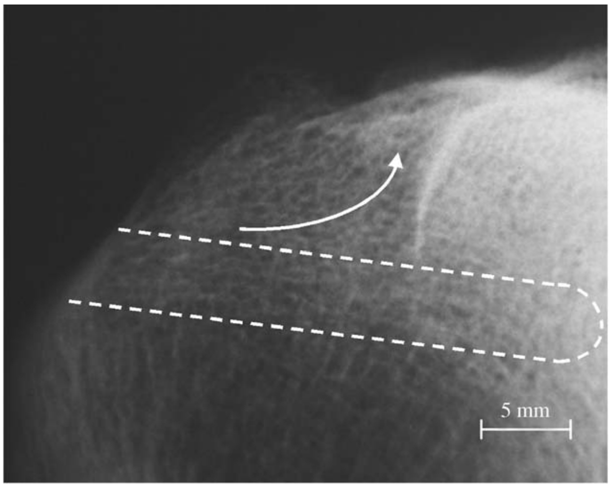

Figure 8 Change in the orientation of bone structure in the sagittal plane of the tibial head. The arrow indicates the diversion of horizontal trabeculae to the vertical orientation at the attachment site of the cruciate ligaments, taking the acting tensile forces. The dashed line indicates the location of the created defect.

These trabeculae are sitting right at the attachment site of the cruciate ligaments, which pull on the tibial plateau exactly in this vertical direction. This particular structure could be loaded in tension on the tibial plateau coming from the two screws, which pull in a slightly horizontal fashion once the embedding is not perfectly horizontally placed on the bone anymore. Tension could lead to a failure of the tibial plateau at exactly this point. On the other side, the lateral side of the plateau carries the tendon of the musculus tibialis cranialis. This reduces the width of the plateau again at the same spot. All together could lead to a weakening of the tibial plateau, which would cause the mentioned angulation of the tibial plateau at loading.

Conclusions

The present study was done as an add-on to the *in vivo* study carried out by Gisep *et al.* [1]. The aim was to investigate the mechanical performance of ceramic bone cements in a highly loaded slot defect in the proximal sheep's tibia. With a low number or samples, it was shown that perfectly filled defects could stand a load of up to 4.5 kN, corresponding to about five times a sheep's body weight. This load could be exceeded by running or jumping or even by walking on uneven ground. The variations in the results were quite low, considered the age of the bones being between 31 and 112 months.

An incomplete filling leading to a gap between the cement and the tibial plateau reduces the loads taken by the bone-implant system dramatically. Only 1000 cycles were done in the dynamic test at loads of about 1/3 of the maximum load taken by the fully filled samples. After this test, cracks were seen in all of the samples, smaller but more in the anterior region, bigger but only a few in the posterior part. The filling of a defect has therefore to be controlled very accurately to give the most support to a highly loaded fracture site.

The optical observation of the whole test run by video cameras has proven to be of great help in this study. The proximal embedding of the tibia probably was the weakest point in the whole study. The Beracryl-block was not allowed to go too far distal for not bridging the defect and on the other hand had to be kept as small as possible to not artificially reinforce the tibial plateau. Fixation of the block to the plateau was not good enough with just the form-fit, therefore, the two screws had to be engaged in the tibial plateau. This led to some fixation of the proximal embedding. The difference in the two deformation measurements (Instron vs. optical system) was mainly due to the strong deformation of the proximal embedding. The video control allowed for subtraction of this error and for a more accurate interpretation of the results.

In the end, this study proved the limited mechanical performance of ceramic bone substitutes. Highly comminuted fractures in loaded areas with a loss in cortical structure have to be adequately stabilised with metallic implants before ceramic bone substitute materials can be injected. Despite their compression strength, which is comparable to cancellous bone or even higher, these materials should only be used as a substitute for cancellous bone or as a filler material in mechanically stable situations.

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Received 19 March
and accepted 29 April 2004