



## The effect of phosphoric and phosphonic acid primers on bone cement bond strength to total hip stem alloys



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### ABSTRACT

Aseptic loosening at alloy-cement interfaces constitutes a main failure mechanism of cemented total hip replacements (THR). As a potential solution we investigated the effect of metal primers containing phosphoric and phosphonic acid on shear bond strength (SBS) of bone cement to THR alloys (CoCrMo, TiAlNb) and pure tin (Sn) substrates (20×8×3 mm). Metal surfaces were modified by polishing or Al<sub>2</sub>O<sub>3</sub> blasting and primer application. Substrates without primer treatment served as references. Cylindrical cement pins (Ø 5 mm) were polymerised onto substrate surfaces and aging (1, 5, 14 and 150 days) was simulated in aqueous NaCl solution (0.9%) before SBS determination and failure mode evaluation.

Regardless of surface roughness and aging time, SBS for THR alloys and Sn was always significantly higher with primer treatment. Compared to untreated reference specimens ( $\leq 0.2$  MPa) SBS values increased even up to 350 fold (TiAlNb, 14 days) or 400 fold (CoCrMo, 5 days). In general, the phosphoric acid containing primer revealed significant higher SBS values on THR alloys compared to the phosphonic acid containing one. Al<sub>2</sub>O<sub>3</sub> blasted specimens showed generally higher SBS values than polished ones with the exception of Sn which showed high SBS values in general. With primer treatment on polished Sn a significant reduction of SBS could not be detected even up to 150 days, whereas THR alloys showed only an SBS improvement in the short term ( $\leq 14$  days). A NaCl-pitting corrosion probably led to an increasing and durable SBS on polished Sn surfaces over time.

Compared to modern THR in clinical practice that shows survival rates of 10, 15, 20 or more years, the receivable bond strength enhancements described in this study appeared to be very short. The improved SBS on THR alloys lasted only a few days before it was lost again. In contrast, the phosphoric acid primer treatment of polished Sn appeared to be very promising and may play a key role in further investigations dealing with the prevention of the stem-cement debonding in THR.

### 1. Introduction

Total hip replacement (THR) is one of the most successful and frequently undertaken elective surgeries (Pivec et al., 2012) and more than 40% of femoral stems worldwide are implanted using cement (PMMA) (Waddell et al., 2010). Stem, cement and bone represent a compact structure containing two different interfaces, namely the stem-cement and the bone-cement interface. For more than 35 years it has been recognised that aseptic loosening constitutes the main failure mechanism of THR (Jasty et al., 1991; Plitz, 1989; Verdonschot

et al., 1998) but no satisfactory solution has been found to date. The identifiable symptomatic loosening of cemented stems at a later stage actually originates soon after surgery in a primary debonding, which is due to an insufficient stem-cement adhesion (Jasty et al., 1991). A cascade of typical implant-loosening effects will only be observed later and are caused by micromovements, surface abrasion, formation of metal and PMMA wear debris, destruction of the cement layer and finally a wear-mediated osteolysis (Buchhorn et al., 2015; Verdonschot et al., 1998). This secondary scenario cannot be avoided but in recent years it has been delayed through the use of specially designed stems.

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These are made of CoCrMo alloys, have a straight-tapered shape and offer highly polished surfaces. After debonding these properties enable the stem to perform fewer interface movements due to a quite inflexible material (Ramaniraka et al., 2000), a low frictional self-locking that results in a transformation of shear stresses into hoop stresses within the stem-cement interface (Verdonschot and Huiskes, 1998; Verdonschot et al., 1998) and minimised abrasive processes (Howell et al., 2004). Efforts to generally avoid or at least to delay the primary cause of failure – the debonding – have scarcely been undertaken.

Some manufacturers attempted to improve the adhesion of PMMA to the metal surface by increasing the surface roughness of their stems. But as soon as the second generation of the Charnley stem with matt surfaces was introduced to the market an increased stem loosening was clinically observed (Dall et al., 1993). A similar effect of aseptic loosening was clinically found by Sporer et al. (1999) and by Collis and Mohler (2002) for Iowa femoral components. Comparable results could be shown for Exeter stems by Howie et al. (1998). One explanation for this increased failure rate was given by Verdonschot and Huiskes (1998), namely that the primary debonding could not be avoided but rather gave rise to a further acceleration of secondary loosening symptoms. Suddenly the roughened stems acted like rasps wearing down the already weakened cement layer (Mumme et al., 2008; Sporer et al., 1999; Verdonschot and Huiskes, 1998).

Subsequent attempts aimed to increase the chemical adhesion between PMMA and metal using adhesive layers comprising silane/silica coatings on implant surfaces (Fischer et al., 2001; Mumme et al., 2008), for example. Although these coatings can at least delay cement hydrolysis, which appears in part to be responsible for premature debonding within the human body (Mumme et al., 2008), a clinical breakthrough has not yet been observed.

A closer look at dental medicine reveals that bonding of organic resins to metal surfaces has a long history in prosthodontic dentistry (Taira and Imai, 1995). Adhesion-promoting monomers were introduced back in the 1970 s and have continuously been improved (Anbar and Farley, 1974; Ikemura et al., 2012). Such monomers can bear phosphoric or phosphonic acid groups in combination with methacrylate substituents and may consequently act as an adhesion promoter between oxidic structures on non-precious metal surfaces and resin composites (Ikemura et al., 2006). To make this significant bond increase available for precious metals (e.g. gold crowns) as well, electroplating with Sn has been established (Swartz et al., 2000), since Sn is recognised as self-passivating (Stirrup and Hampson, 1976) and that allows the adhesive-promoting monomers to interact with oxides again.

CoCrMo and TiAlNb alloys typically used for THR have self-passivating properties as well due to their ability to form stable oxide layers on their surfaces. These alloys may therefore have a predisposition for enhancing bond strength of cement in combination with primers containing acidic monomers. Consequently the aim of the present study was to analyse the effect of phosphoric and phosphonic acid containing metal primers on bond strength of bone cement adhered to commonly used THR alloys. The following questions were addressed: (1) Do phosphoric and phosphonic acid containing metal primers enhance the bond strength to CoCrMo and TiAlNb alloys? (2) Do Sn surfaces serve as a possible alternative for improving bond strength with these metal primers? (3) Does surface roughness have an effect on primer-dependent bond strength? (4) Are there differences between phosphoric and phosphonic acid containing primers? And finally, (5) are these metal primers able to avoid bond strength being weakened by hydrolytic cement degradation?

## 2. Materials and methods

Details of all the materials used in this study are provided in Table 1.

### 2.1. Metal substrates

Casted Co-28Cr-6Mo alloy according to ISO 5832-4 (Waldemar Link GmbH & Co. KG, Hamburg, Germany), forged Ti-6Al-7Nb according to ISO 5832-11 (Zimmer Inc., Warsaw, USA) as well as pure Sn ( $\geq 99.9\%$ ) according to DIN EN 610 (Cmet GmbH, Wendelstein, Germany) were included. Eight substrates from one batch of each metal with dimensions of  $20 \times 8 \times 3$  mm were machined by water-jet cutting from solid materials to avoid any thermal impact. Roughnesses on one of the  $20 \times 8$  mm areas as the testing surface were consecutively produced with reference to polished CoCrMo and blasted TiAlNb THRs. According to the experimental matrix (Table 2) surfaces were repeatedly modified by water-cooled mechanical polishing (4000 grade SiC paper on plate grinder 400CS, Exact GmbH, Norderstedt, Germany) as well as blasting with  $\text{Al}_2\text{O}_3$  (type F45 according to FEPA-Standard 42-1:2006; Hafra GmbH, Assling, Germany). Surface roughness of blasted substrates (Table 3) was adjusted to reference values received from an untouched cementless THR made from TiAlNb (CLS<sup>®</sup>, Zimmer Inc., Warsaw, USA) using an optical profilometer (MicroProf, Fries Research & Technology GmbH, Bergisch Gladbach, Germany). Surfaces, therefore, ranged within upper roughnesses used in cemented THR in the past ( $R_a \approx 5.1 \mu\text{m}$ ) (Choi et al., 2010; Damron et al., 2006; Ebramzadeh et al., 2004). Different hardnesses of metals were therefore considered by material-specific, jet-pressure adjustments, whereas nozzle distance was kept as a constant (30 mm).

### 2.2. Metal primer and bone cement applications

All procedures were performed under an air-conditioned laboratory atmosphere ( $15^\circ\text{C}$  and 52% humidity). The GC Metal Primer II (GC Europe N.V., Leuven, Belgium) containing a thiophosphoric acid methacrylate (Fonseca et al., 2009; Taira and Imai, 1995) and the Sebond Smart Primer (Schütz Dental GmbH, Rosbach, Germany) containing a phosphonic acid-substituted silicon methacrylate (Leyhausen et al., 2005) were chosen. Schematic chemical interactions of both primers with metal oxide surfaces and PMMA adherence can be taken from Fig. 1. Both primers were applied to substrate surfaces with a brush for 5 s and subsequently dried with compressed dry air for 5 s before cement application. Substrates without any primer treatment served as a reference to the primer-treated metals. By means of a fixation device from the UltraTester<sup>™</sup> system (Ultradent Inc., South Jordan, Utah) polyethylene tubes (inner diameter=5 H7) as bonding mould inserts were centrally placed onto substrate surfaces immediately after primer application (Fig. 2). Through these tubes a low-viscosity, 2-component bone cement of one batch as typically used for THR (Palacos-R, Heraeus Medical GmbH, Hanau, Germany) was inserted and was pressed for 20 s onto substrates with a 30N-stamp. The pre-chilled cement ( $T=7^\circ\text{C}$ ) was mixed as recommended by the manufacturer for 30 s. After complete curing for 15 min at  $15^\circ\text{C}$  both the fixation device and the bonding mould inserts were carefully withdrawn from the specimen leaving a cylindrical cement pin on the surface (Fig. 2). To simulate aging processes within body environment all finished specimens were stored at  $T=37.5^\circ\text{C}$  in NaCl solution (0.9%) for 24 h, 5, 14 and 150 days.

### 2.3. Shear bond strength

As in clinical practice the stem/cement interface is mainly exposed to stresses caused by shear forces in the non-debonded condition (Mumme et al., 2008; Plitz, 1989), we decided to determine the shear bond strength (SBS) of cement pins fixed to metal substrates using a materials testing machine (Type Inspekt 20, Hegewald & Peschke GmbH, Nossen, Germany). For the loading setup we used an established shearing-off device (Jung et al., 1999) containing a wedge-shaped chisel that was driven against the pin bottom at a crosshead speed of 2.5 mm/min (Figs. 3–5). Since the bonding area was known

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