

Elsevier Editorial System(tm) for Sensors & Actuators: A. Physical  
Manuscript Draft

Manuscript Number:

Title: Low-firing thick-film piezoresistive sensors for medical instruments

Article Type: Special Issue: Eurosensors 2010

Section/Category: Materials and Technology

Keywords: Medical operations; total knee arthroplasty; thick-film force sensors; medical alloys

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Abstract: In this work, a low-firing thick-film materials system allowing fabrication of piezoresistive sensors on surgical alloys is presented in detail, with application to a force-sensing surgical instrument. The system comprises a series of individual thick-film dielectric, conductor, resistive and overglaze compositions based on a lead borosilicate glass matrix. The moderate achieved firing temperature, around 625°C, greatly increases compatibility with metallic substrates, allowing the use of high-strength medical alloys with low thermal degradation. Specific fillers for the dielectric layers increase adhesion on steel substrates and allow thermal matching to austenitic and ferritic / martensitic steels, as well as titanium alloys, and preliminary work demonstrating reactive stabilisation of the dielectric layer to achieve an even wider process window is also shown. Finally, the functionality of this materials system is successfully demonstrated here by implementing it into a previously developed ligament-balancing force sensor for total knee arthroplasty (TKA) [1].

## Low-firing thick-film piezoresistive sensors for medical instruments

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COVER LETTER

30.9.2010

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Dear Editor,

Please find attached the files for the paper entitled “Low-firing thick-film piezoresistive sensors for medical instruments” authored by Thomas Maeder, Caroline Jacq and Peter Ryser, which is an extension of the extended abstract presented at Eurosensors XXIV as contribution #3419.

This contribution reports the formulation of low-firing (<650°C) glass-based thick-film materials systems that allow deposition of piezoresistive bridges onto high-strength stainless steels without excessive degradation of the substrate mechanical properties, and especially avoiding the austenitic-martensitic phase transitions that would be destructive for the thick-film layers. Preliminary work demonstrating reactive stabilisation of the dielectric layer to achieve an even wider process window is also shown. With further improvements / refinements, we expect to be able to achieve even lower process temperatures (500-600°C), further decreasing alteration of the properties of high-strength steels and allowing deposition on Ti and even some Al alloys. In a second part of the work, the aforementioned materials system is successfully applied to a previously developed complex medical force sensor, used for balancing ligament tension in knee operations.

Please let me know in case you find anything missing during the review of the paper.

Best regards

Thomas Maeder

*(Revised + extended version of paper # 3419 presented at Euroensors XXIV conference in Linz)*

# Low-firing thick-film piezoresistive sensors for medical instruments

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## Abstract

In this work, a low-firing thick-film materials system allowing fabrication of piezoresistive sensors on surgical alloys is presented in detail, with application to a force-sensing surgical instrument. The system comprises a series of individual thick-film dielectric, conductor, resistive and overglaze compositions based on a lead borosilicate glass matrix. The moderate achieved firing temperature, around 625°C, greatly increases compatibility with metallic substrates, allowing the use of high-strength medical alloys with low thermal degradation. Specific fillers for the dielectric layers increase adhesion on steel substrates and allow thermal matching to austenitic and ferritic / martensitic steels, as well as titanium alloys, and preliminary work demonstrating reactive stabilisation of the dielectric layer to achieve an even wider process window is also shown. Finally, the functionality of this materials system is successfully demonstrated here by implementing it into a previously developed ligament-balancing force sensor for total knee arthroplasty (TKA) [1].

Keywords: Medical operations; total knee arthroplasty; thick-film force sensors; medical alloys

## 1. Introduction

Smart surgical instruments including load-sensing technology and allowing feedback to the surgeon, robotic surgery and haptics lead to better repeatability [1,2], safety and success rate of surgical operations. In addition, load-sensing implants and various robotic devices may constitute a useful tool in monitoring patient

recovery, aiding re-education and rehabilitation [3]. Compared to polymer-based devices, mineral thick-film materials feature good structural and chemical stability, and allow hermetic packaging, which translates into stable implant behaviour and good sterilisability (autoclave or dry heat) for instruments.

On grounds of cost and mechanical reliability, biocompatible metallic structural materials such as stainless steels and Ti alloys [4-6] are often preferred over ceramics for tools and load-bearing implants. However, although thick-film force / pressure sensing on metals has been well-known for a long time [7-10], the high standard 850°C thick-film firing temperature is incompatible with most high-strength medical alloys. Ti and its alloys experience strong surface oxidation [1,11] that would require complete enamelling or post-deposition machining, which is not very practical. Our first experiments with well-available high-strength precipitation-hardening martensitic stainless steels failed due to martensitic-austenitic-martensitic phase transitions (Fig. 1, [12]) that lead to mechanical destruction of the overlying layers. For such steels, process temperatures should ideally lie at 550°C to avoid any strength reduction due to annealing effects, but around 600°C would be tolerable [12,13]. Phase transformation and annealing problems may be (at least in part) circumvented by the use of precipitation-hardened [14] or nitrogen-alloyed solution-hardened [1] austenitic stainless steels, the latter of which, besides mechanical strength, also features improved corrosion resistance [4,14]. However, high-nitrogen steels are expensive to machine, and their high coefficient of thermal expansion (CTE) gives rise to problematic compressive stresses; in our first device intended to be fabricated with commercial thick-film materials only [1], a basic dielectric-conductor-resistor buildup (Fig. 2) was still possible, but the addition of crossovers (i.e. another dielectric-conductor sequence) inevitably led to delamination (Fig. 3). This prompted the introduction of a low-stress crossover dielectric formulated with a low-melting glass. However, yield and reliability were still somewhat problematic due to the high compressive stresses in the main dielectric.

## FIGURE 1

In order to solve these problems and additionally allow the use of high-strength (and lower CTE) Ti alloys or martensitic steels, we endeavoured to develop a reliable thick-film materials system featuring low

(<650°C) firing temperature [16,17] and adaptability to a large palette of alloys. A typical piezoresistive thick-film materials sequence, depicted on Fig. 2, mainly comprises dielectric, conductor and resistor materials with the optional addition of an "overglaze" glassy protection layer, and must optimise many aspects, such as CTE compatibility with the metallic substrate, adherence and chemical compatibility among layers in general and with the steel, strain-sensing properties of the resistors, while achieving good reliability, i.e. low process sensitivity, good reproducibility and long-term stability. This work reviews the development of a first-generation low-firing system for piezoresistive sensing on metallic substrates, and details its successful application to the TKA sensor.

## FIGURE 2

### 2. Low-firing thick-film materials

#### 2.1. Glasses

Except commercial compositions, all formulations in this study are based on the same two lead borosilicate glasses (V6 & V8; data given in Table 1) used in our previous studies on low-firing dielectrics & resistors [16-18]. They feature a high PbO content to obtain lower processing temperatures and an extra 2% Al<sub>2</sub>O<sub>3</sub> added to suppress crystallisation [19]. For dielectrics or resistors, the ideal firing temperature lies ca. 100°C above the densification temperature of the glass alone, namely around 600°C for V6-based materials and around 500°C for V8-based ones. In this study, all dielectrics base on V6, with lower-melting V8 used for fritted conductors and overglazes.

#### 2.2. Dielectric stack

Given the aforementioned requirements, it appears rapidly (Fig. 2b) that the dielectric, which fulfills many functions, is by far the most critical and complex element; it makes chemical contact with all other layers, is responsible for the critical mechanical bonding to the steel and, as it is by far the most voluminous material, a good CTE matching is mandatory. These requirements may be partially conflicting and therefore problematic to fulfil with a single material. For instance, additives to improve adhesion and CTE matching

may harm chemical compatibility with the resistor or even the insulating properties of the dielectric. However, these different functions may be carried out by different layers, as illustrated in Fig. 2b, indicated in Table 2 and detailed hereafter.

### **TABLE 1**

### **TABLE 2**

#### *2.2.1. Adhesion layer*

Preliminary studies showed poor adhesion of basic filled-glass dielectrics to stainless steels, a situation only somewhat improved by prior oxidation of the substrate. This is thought to be due to oxygen depletion at the interface, which was countered by filling the first dielectric layer with  $\text{Fe}_2\text{O}_3$ , which provides a source of oxygen by partial reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$ . While we did not confirm its supposed mode of action, this solution was found to be an efficient adhesion promoter, failure nearly never occurring at the metal-dielectric interface [17].

#### *2.2.2. Main layer – CTE adjustment*

The CTE of this layer was adjusted by adding suitable fillers to the glass matrix, which provide mechanical stabilisation of the dielectric as well. This "main" dielectric composition was applied to crossovers as well. As fillers, we used quartz for the ferritic and martensitic stainless steels (CTE  $\sim 11$  ppm/K), and cristobalite for the austenitic ones (CTE  $\sim 17$  ppm/K).

#### *2.2.3. Chemical compatibility layer*

$\text{SiO}_2$  (quartz & cristobalite) fillers allow tuning of dielectric CTE, but tend to dissolve into the glass. As dissolution of the silica fillers into the high-lead V6 glass was found to lead to poor compatibility with both V6-based resistors and the commercial low-firing resistive composition ESL 3114 (ElectroScience Laboratories, USA; thought to be based on a similar glass based on its materials safety data sheet), a chemical compatibility layer was introduced; it consists of V6 filled with alumina powder, which has poor

CTE matching to the steel, but is more inert towards the resistor. For Ti alloys (CTE ~8 ppm/K), this composition can be also used as a main layer.

### *2.3. Other layers*

Deposition of conductors and resistor layers followed the modified scheme developed in our previous study [17]. First, the solder pads and main conductive tracks are deposited and dried using a layer of commercial ESL 9912 Ag conductor, which gives good adhesion and solderability but does not constitute a good resistor termination. Then, the first of two crossover dielectric layer (if required) is deposited and co-fired at 625°C with the 1<sup>st</sup> conductor. After this, the 2<sup>nd</sup> crossover dielectric layer is deposited and dried, followed by a commercial resistive composition, ESL 3114 (10 kΩ) and again co-firing at 625°C. For the resistor terminations, a custom conductor consisting of Ag powder (90% vol.) and V8 glass frit (10% vol.) is printed and post-fired at 500°C. This 2<sup>nd</sup> conductor layer comprises the resistor terminations, acts as "reinforcement" for the solder pads and also as the "top" conductor for the crossovers. Finally, a protective overglaze of straight V8 glass is deposited and fired at 425°C, ending the "high-temperature" processing steps, followed by a parylene coating to guard against possible leaching of lead from the glasses.

### *2.4. Reactive stabilisation of dielectric – exploratory work*

The low-firing dielectrics described in the previous section still do suffer from one shortcoming: a relatively delicate balance between glass type, filler loading and processing temperature must be struck, to reliably achieve densification, yet avoid "swimming" and conductor contamination by glass. This stems from our dielectrics essentially belonging to the "filled" category [20,21], i.e. consisting of a glass and a nominally nonreactive filler that, besides tuning the CTE, mechanically stabilises the glass; apparently, the degree of SiO<sub>2</sub> or Al<sub>2</sub>O<sub>3</sub> dissolution when firing near 600°C is insufficient to allow strong stabilisation, in contrast to modern reactive / crystallising dielectrics that exhibit good re-fire stability.

In a previous work [22], we showed such reactive stabilisation was possible with lead borosilicate glasses by adding TiO<sub>2</sub> fillers; the system is stabilised both by PbTiO<sub>3</sub> formation (increase of filler volume fraction) and the resulting PbO depletion of the glass matrix. However, PbTiO<sub>3</sub> has very low CTE and is therefore not ideal as a filler on high-CTE substrates. Nevertheless, other "acidic" oxides such as MoO<sub>3</sub> have shown to

react similarly to  $\text{TiO}_2$  with high-lead glasses, yielding higher-CTE  $\text{PbMoO}_4$  [22]. Therefore, the possible reactions between V6 glass with 5.2% mass  $\text{MoO}_3$  (Sigma-Aldrich M0753,  $\geq 99.5\%$ ), formulated as thick-film pastes and fired on alumina substrates, were examined by X-ray diffraction.

The results are shown in Fig. 4 for single typical thick-film firing cycles, with a 10 min dwell at the indicated temperature. The system is very reactive; significant  $\text{PbMoO}_4$  (wulfenite) formation is already evident at the lowest  $525^\circ\text{C}$  firing temperature, and the original  $\text{MoO}_3$  (molybdite) peaks essentially disappear for firing temperatures  $\geq 575^\circ\text{C}$ . The experiment was repeated for closely related  $\text{WO}_3$  (Sigma-Aldrich 95410, 17% mass in V6 paste), yielding  $\text{PbWO}_4$  (stolzite) as a reaction product. Qualitatively,  $\text{WO}_3$  is somewhat less reactive than  $\text{MoO}_3$ , disappearing in the  $600\text{--}625^\circ\text{C}$  firing temperature range, which is expected from its higher melting point and lower volatility.

These results demonstrate that reactive stabilisation of the developed low-firing dielectrics by suitable additions is in principle possible, even at moderate firing temperatures, potentially yielding materials with a wide process window and excellent re-fire stability akin to that of modern commercial  $850^\circ\text{C}$ -firing dielectric compositions.

#### FIGURE 4

### 3. Application to TKA ligament-balancing force sensor

The steel mechanical structure of the sensor and testing jig / procedure were the same as before [1], with the new low-firing thick-film materials system (austenitic steel variant) replacing the mainly commercial materials, and the conductor layout modified to reflect the abovementioned new layer sequence (see 2.3). A picture of the resulting sensor is given in Fig. 5a, with the measurement results (force & X position) for one plate shown in Fig. 5bc.

#### FIGURE 5



In line with our prior results [16,17], good adhesion and reliability was observed for the new version of the TKA ligament-balancing sensor (two sensing platforms, one for each condyle; each platform fitted with three force sensors allowing determination of total force and XY position). After calibration, testing at several positions (labelled *efgh* on Fig. 5bc) resulted in good matching between measured and applied total force (Fig. 5b), with a low position error for sufficiently large forces (Fig. 5c).

#### **4. Conclusions and outlook**

A complete low-firing thick-film materials system for ferritic / martensitic and austenitic steels was developed. The materials were formulated / selected to allow firing at 625°C, which was found to be a good compromise between low processing temperature and good stability. This firing temperature allows application to high-strength steels, as it avoids problems with phase transitions and reduces softening due to annealing. Also, degradation by oxidation is considerably reduced, possibly leading in the future to application on titanium alloys as well. This new materials system was applied successfully to our previously developed ligament-balancing sensor for TKA, demonstrating its potential for fabrication of complex piezoresistive sensor cells.

Preliminary results in this work show that the developed materials may be further improved to achieve even better processability, which will be the object of further studies, together with switching to lead-free compositions, which recently have yielded promising results [24,25].

#### **Acknowledgements**

The authors gratefully acknowledge financial support from the Swiss National Science Foundation (SNSF) through the NCCR CO-ME II programme, the dilatometry measurement by Dr. Sonia Menot, the help of Mr. Matthias Garcin in preparing the samples, and the supply by the University of Bern ARTORG centre of the mechanical structure and testing jig.

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### Biographies

**Caroline Jacq** graduated with a Master thesis in materials science from the Institut Supérieur des Matériaux du Mans (ISMANS), and is working as a researcher at the Ecole Polytechnique Fédérale de Lausanne (EPFL) since 2001. Her main research interests concern the development and study of novel lead-free thick-film materials systems and their application to medical devices.

**Thomas Maeder** graduated at the Ecole Polytechnique Fédérale de Lausanne (EPFL) in materials science, and continued with a PhD in piezoelectric thin films, then a post-doc at IBM Rüschlikon in single-crystal conductive oxides. He now heads the thick-film

technology group at the EPFL, where current areas of interest are thick-film and LTCC technology for advanced sensor, packaging, biomedical and harsh-environment applications.

**Peter Ryser** received a Master degree in Physics (University Neuchâtel 1979), a PhD in applied Physics (University Geneva 1985) and a Masters Degree in Corporate Management (Lucerne 1993). His professional background includes several R&D activities. From 1990-1998 he was the head of research at Siemens Building Technologies. Since 1999 Peter Ryser is Professor at the Swiss Federal Institute of Technology EPFL in Lausanne and act as a director for the micro engineering section.

#### FIGURE CAPTIONS

**Fig. 1.** Dilatometry (ca.  $\pm 100$  K/min) performed on cylindrical 1.4542 (USA: 17-4PH) steel. M/F = martensite/ferrite, initial state; A = austenite.

**Fig. 2.** (a) typical thick-film stack; (b) dielectric buildup detailing the functions of the different layers.

**Fig. 3.** Delamination problems encountered with commercial materials when introducing crossovers.

**Fig. 4.** Powder X-ray diffractogram of V6 + 5% mass MoO<sub>3</sub> vs. firing cycle. M = MoO<sub>3</sub> peaks; PM = PbMoO<sub>4</sub> peaks.

**Fig. 5.** (a) sensor platforms screen-printed with new low-firing thick-film materials; (b) measured force and (c) X position error after calibration.

#### TABLE CAPTIONS

**Table 1.** Composition of both glasses. Percentages refer to PbO+B<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>. Approximate densification temperature for 10 min at peak.

**Table 2.** Dielectric multilayer buildup (all layers fired at 625°C). † Cristobalite for austenitic steels (here) or quartz for ferritic / martensitic.

Figure 1  
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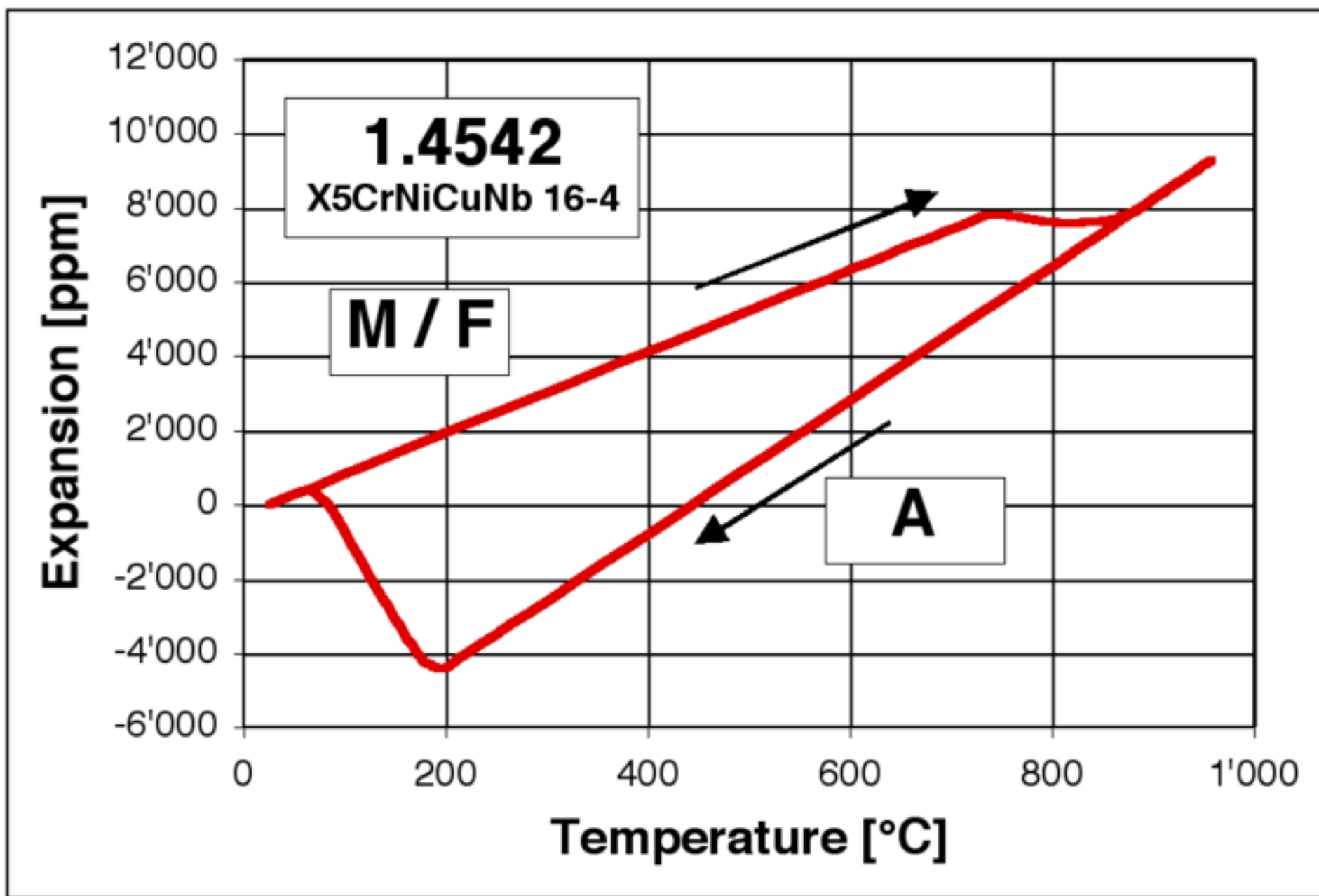
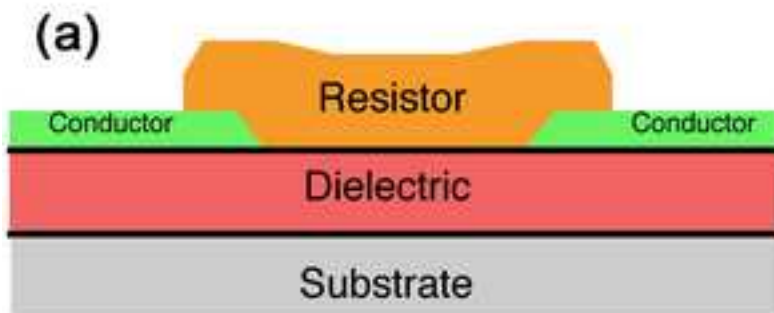


Figure 2  
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<b>Measuring bridge</b>	Conductor & resistor layers (+ overglaze)
<b>Dielectric 3 : compatibility</b>	Chemical compatibility with conductors & resistors
<b>Dielectric 2 : main layer</b>	Good CTE matching Electrical insulation Refire stability (no re-melting)
<b>Dielectric 1 : adhesion</b>	Adhesion promoters + good CTE matching
<b>Substrate</b>	Medical alloy – steel

(b)

Figure 3  
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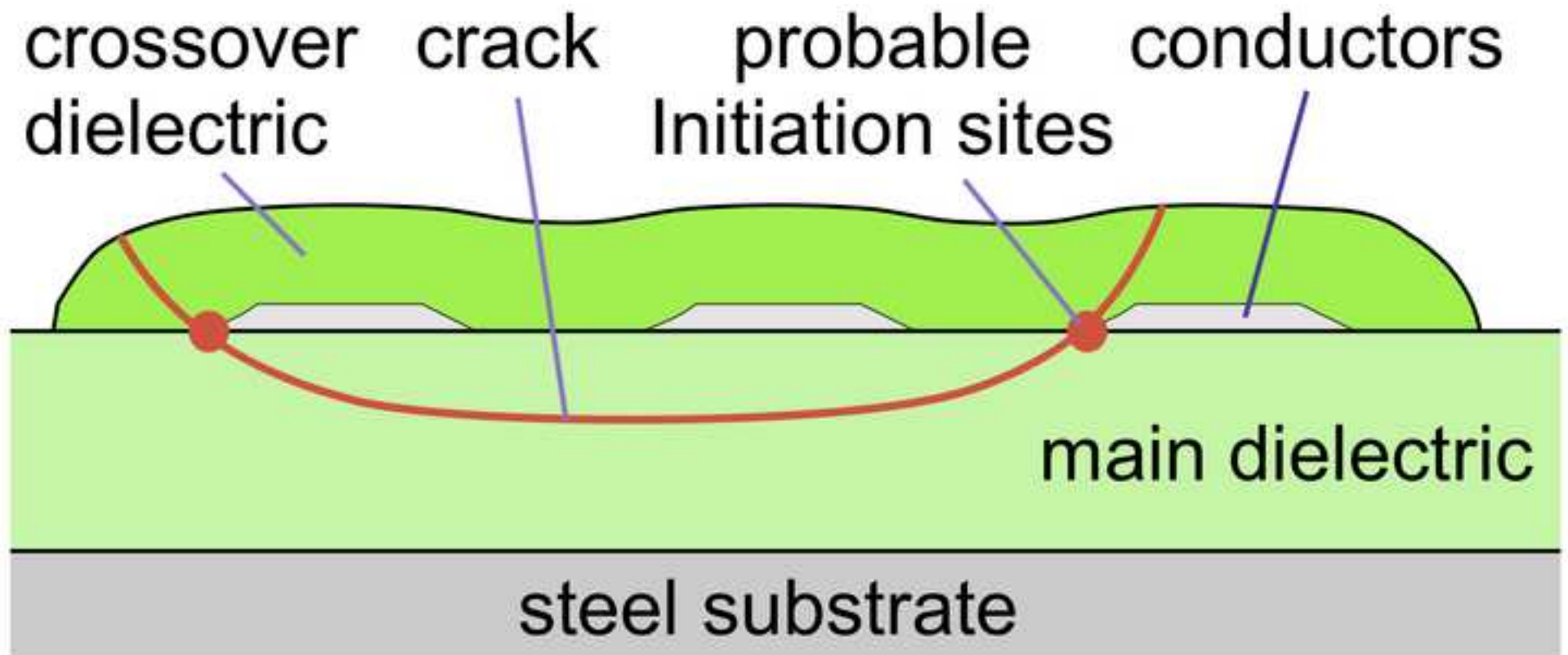


Figure 4  
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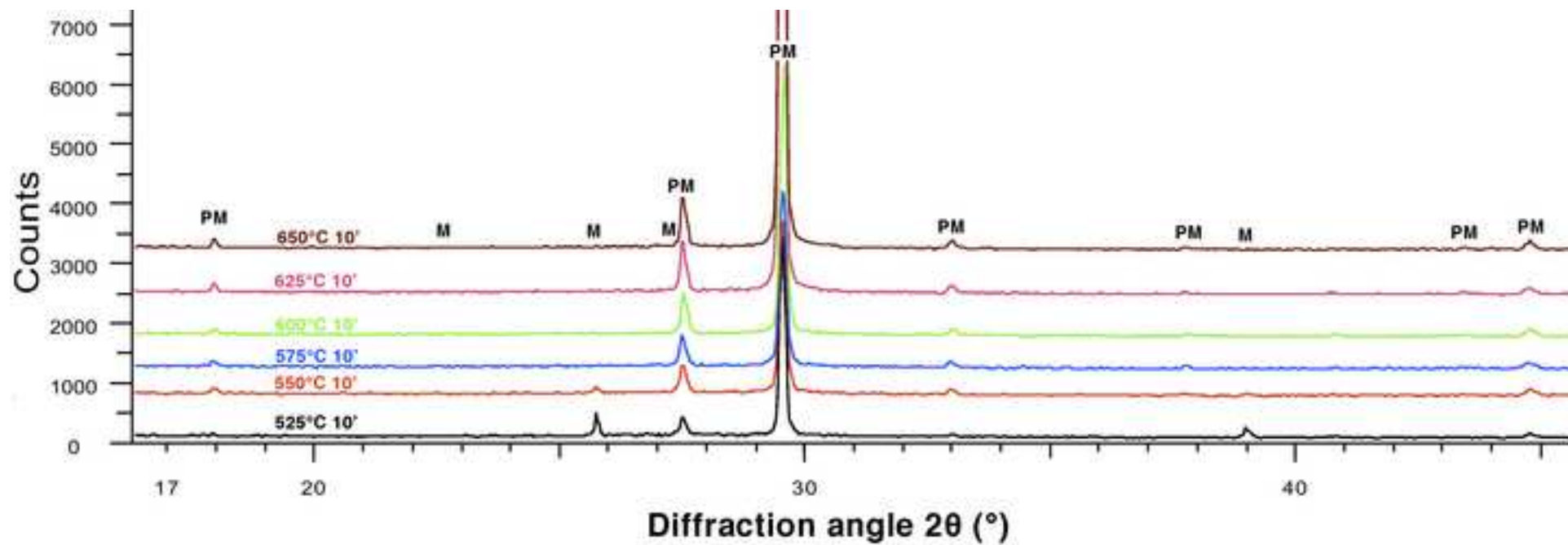
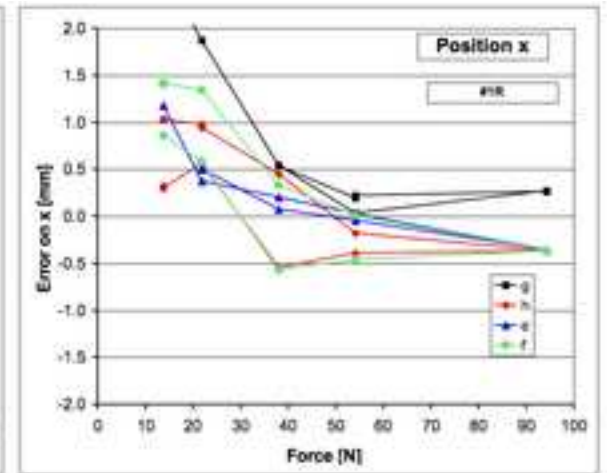
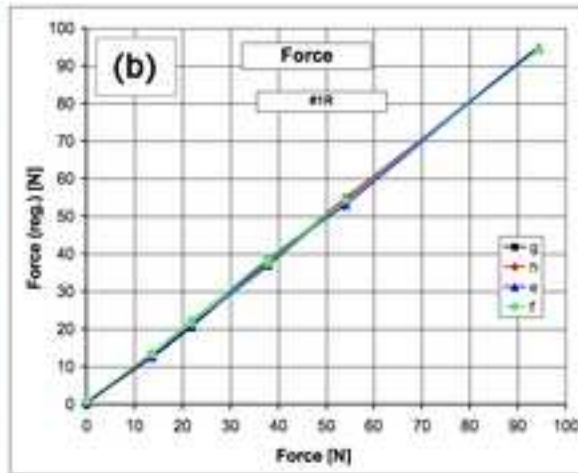
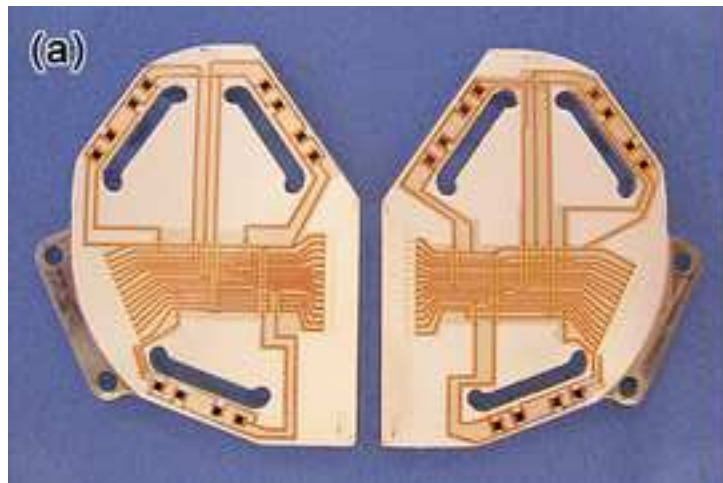




Figure 5  
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(Revised + extended version of paper # 3419 presented at Euroensors XXIV conference in Linz)

## Low-firing thick-film piezoresistive sensors for medical instruments

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### Tables

**Table 1. Composition of both glasses. Percentages refer to  $\text{PbO}+\text{B}_2\text{O}_3+\text{SiO}_2$ . Approximate densification temperature for 10 min at peak.**

Glass		V6	V8
PbO	(%mass)	75	85
B <sub>2</sub> O <sub>3</sub>	(%mass)	10	10
SiO <sub>2</sub>	(%mass)	15	5
Al <sub>2</sub> O <sub>3</sub> addition	(%mass)	2	2
Densification temperature (glass)	(°C)	500	400
CTE	(ppm/K)	≈9.0	≈10.5

**Table 2. Dielectric multilayer buildup (all layers fired at 625°C). † Cristobalite for austenitic steels (here) or quartz for ferritic / martensitic.**

Layer (# prints)	Thickness (μm)	Glass (% vol.)	Filler (% vol.)
Crossover (2x)	20	(= main)	(= main)
Compatibility (1x)	20	50 V6	50 Al <sub>2</sub> O <sub>3</sub>
Main (2x)	40	40 V6	60 SiO <sub>2</sub> †
Adhesion (1x)	17	75 V6	25 Fe <sub>2</sub> O <sub>3</sub>