Piezoresistive properties of low-firing temperature thick-films on steel sensors

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Abstract

Thick-film materials are very advantageous for piezoresistive pressure and force sensors because of ease of processing, reliability and low cost. The standard substrate material used with thick-film technology for sensing is alumina, but its elastic modulus is high and its strength rather low. Steels offer better mechanical properties and permit assembly without an elastomer seal, which is required for pressure sensing in severe conditions. In order to use the steel as substrate, the standard firing temperature of thick-films has been decreased. In previous studies, we have developed and characterized 2 low-firing thick-film systems (dielectrics, resistors and conductors) compatible with austenitic and ferritic materials. We have formulated these systems to achieve to chemical and thermal expansion compatibility. Other parameters like adherence, soldering properties and process, have been optimized too in order to be adapted on high-performance sensors.

In this work, we will present the characterization of 2 steel sensors based on low fired thick-film technology: a high-performance pressure sensor based on high-strength steel substrate chemically similar to the ferritic steel, and a force sensor used in surgical operation of total knee arthroplasty (TKA) based on a medical alloy comparable to the austenitic steel.

Key words: thick film system, high strength steel, pressure sensors, low-temperature firing.

1. Introduction

Pressure and force sensors based on thick-film (TF) technology deposited on ceramic substrates have found success due to their low production cost. However, alumina is not optimal for piezoresistive sensing applications, as it is brittle, its elastic modulus is high and its strength rather low [i] compared to the high strength steels and the austenitic steels. Additionally, the applications of this kind of sensors remain limited because their assembly requires elastomer seals in severe conditions. Stainless steels [ii] potentially offer much better mechanical properties, as well as hermetic assembly by welding. Metallic materials also offer advantages such as toughness and machinability.

However, the high temperatures associated with commercial thick-film processing (850°C) are not compatible with high performance steel and undergo degradation of mechanical properties due to annealing or phase transformations incompatible with the presence of thick-film layers. In order to avoid this problem, a potential solution resides in applying special steels whose mechanical properties are not degraded at 850°C. Another solution would be to develop a thick-film system with a lower firing temperature, ideally below ca. 650°C, a temperature still compatible with good strength retention and that avoids phase transformations. Such systems need to be thermally matched to steels, which have a range of thermal coefficients of expansion (TCE) from 11 ppm/K to 17 ppm/K, compared to standard thick-film materials, which are thermally matched to alumina (7 ppm/K).

In previous studies [iii-8], we have developed and studied several such low-firing systems based on low-melting lead borosilicate glasses, where the materials were formulated to achieve compatibility with steels which cover a wide range of CTE (11…17ppm/K). CTE matching (which is important mainly for the insulating dielectric) was successfully achieved by adjusting the glass composition, the filler and the loading. Adherence and soldering properties have also been examined with a suitable layer and by adapting the thick film process.

In this work, we present the results of applications of previous developed low-firing thick films on two steels: a high strength steel, chemically similar to the ferritic steel, for a high-performance pressure sensor and an austenitic steel comparable to a medical alloy for a force sensor used in surgical operation of total knee arthroplasty (TKA) based on the austenitic steel. These results have been compared to sensors obtained on special high-temperature resistant steel using the commercial 850°C-firing thick film system.

2. Experimental

Substrates

The substrates used for the high strength pressure sensors are: a) a special high-temperature resisting steel denoted HT, b) 17-4 PH / EN 1.4542 and c) Special Metals A286. HT is a ferritic (TCE = 11 ppm/K) steel, which does not undergo phase transformation or soften significantly upon exposure to 850°C, which makes it compatible with the standard thick film process. It has been used as a reference material, but it is very expensive to machine and therefore not compatible with high volume production. 17-4 PH and A286, on the other hand, require a lower temperature thick-film process, and are compatible with maximum firing temperatures of ca. 650°C and 750°C respectively. 17-4 PH is a martensitic (11 ppm/K) precipitation hardening steel having a very high strength and commonly used in thin-film and glued strain gauge sensors. Two sources for 17-4 PH were used in this study: 1) commercial bar where sensors were machined and 2) parts by metallic injection molding (MIM, Figure 1). A286 is a special austenitic (17 ppm/K) precipitation hardening steel, and is solution treated for 1h30 @ 900 °C, quenched and hardened for 16h @ 730°C prior to thick-film deposition. This thermal treatment can also be used to improve the adhesion of the dielectrics by adequate pre-oxidation of the steel.
For the pressure sensor for the TKA operation, an austenitic medical alloy, similar as the standard steel 1.4435, was used.

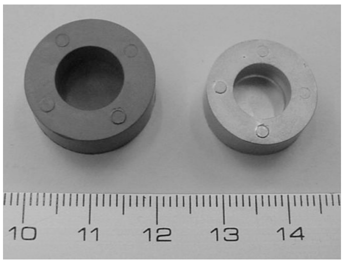

Figure 1: MIM green and sintered 17-4 PH cells (top and bottom)

Thick-film systems

Three TF systems have studied on two temperature ranges (table 1). A classical TF system, 850°C firing and using only commercial materials, comprised the following compositions: 1a) Electro Science Laboratories (ESL) 4916 dielectric, used on steel as an interlayer to decrease interfacial stresses, 1b) Heraeus (Her) GPA 98-029, used as the main dielectric, 2) ESL 9635B (Ag:Pd 3:1 conductor) and 3) DuPont (Du) 2041 (10 kOhm resistor).

Both other TF systems (Figure 2) consist of several dielectrics fired separately at a peak temperature of 625°C, with a 10 min dwell. All dielectrics are based on the same lead borosilicate glass ("V6") used in our previous studies [iv-8]: 75% PbO + 10% B_2O_3 + 15% SiO_2 (mass%) with 2% Al_2O_3 added to inhibit crystallisation [9].

The first dielectric layer is composed of 25% vol. Fe_2O_3 (Alfa Aesar 014680, 99.945%, <5 µm) as an adhesion promoter. The 2 following dielectric layers were filled with 60% vol. cristobalite (Quarzwerke, Sikron cristobalite flour SF8000 D50:2.5µm particle size) for the austenitic steels and with 60% vol. quartz (Nanostrutured & Amorphous Materials Inc. Quartz- SiO2, D50:2.5-3 µm particle size) for the ferritic steels. The filler powder (quartz or cristobalite) serves to dimensionally stabilise the dielectric, and to control its TCE. A top dielectric, filled with 50% vol. Al_2O_3 (Alfa Aesar, aluminium oxide alpha, 99.99%, 1 µm) was applied in order to improve chemical compatibility with the piezoresistor material.

Electro Science Laboratories (ESL) 3114 resistance (10 kOhm, 625°C firing for porcelain enamelled steel) and ESL 9912A Ag conductor used for wires and pad connections were also co-fired at 625°C/10 min. In order to avoid undesirable high-resistive zones near the terminations, the conductive material serving this purpose was post-fired at a lower temperature (500°C). This conductor consists of Ag powder (Nanostrutured & Amorphous Materials Inc. 99.9%, APS 30 nm) with a very low-melting glass binder ("V8", 85% PbO + 10% B_2O_3 + 5% SiO_2, mass%, + 2% Al_2O_3 [3-8]).

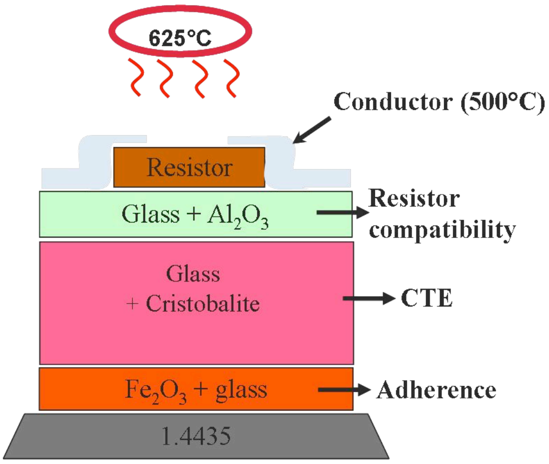

Figure 2: Thick-film sequence with low firing temperature

Table 1: List of sample series and firing temperatures

Substrates	Dielectrics	Conductor & Resistor	Resistor termination
HT	Commercial 850°C	ESL9635B 850°C	Du2041 850°C
A286	$25Fe_2O_3V6$ V6C60 $50Al_2O_3V6$ 625°C	ESL9912 A & ESL3114 625°C	90AgV8 500°C
17-4-PH	$25Fe_2O_3V6$ V6Q60 $50Al_2O_3V6$ 625°C	ESL9912 A & ESL3114 625°C	90AgV8 500°C

Samples

The pressure sensors are based on a monolithic membrane structure (Figure 3), Fabricated by machining from a bar or by MIM. The membrane diameter is 9.35 mm and the thickness is ca. 0.9 mm for the HT steel and A286 steel sensors, and 0.8 mm for the 17-4 PH steel. The nominal pressure, for ~100 MPa stress, is 40 bar for the cells 0.8 mm thickness and 50 bar for the cells 0.9 mm thickness. The thick-film conductors and resistors form a piezoresistive bridge on these membranes. The cells have been measured with a DH-Budenberg 540 VHX hydraulic dead weight pressure tester. For each pressure, the signal is first measured under load, and then after unloading, which allows the evaluation of both response and drift. The supply voltage of the cells is 10.0 V, giving a bridge current around 1 mA and a dissipated power around 10 mW.

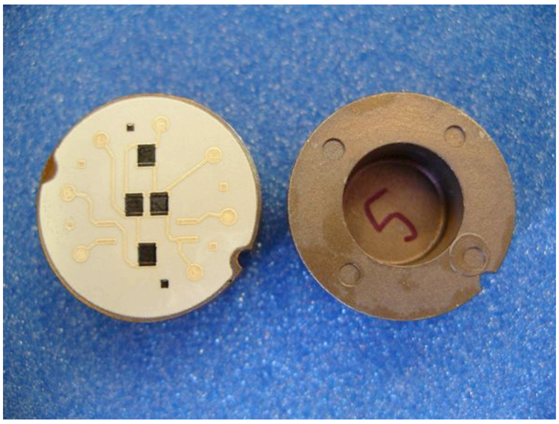

Figure 3: MIM sintered parts (top and bottom)

3. Results

Commercial TF fired @ 850°C and low temperature fired TF (LTTF) fired @ 625°C on HT steel.

Figure 4 and Figure 5 respectively depict the signal and the relative drift (normalised to the response at a nominal stress of 100 MPa, which corresponds here to a pressure of 50 bar). We can note that the response is quite linear and reproducible, and higher for the low temperature TF system (4.52 ± 0.17 mV/V) than for the high temperature TF (HTTF) system (3.55 ± 0.01mV/V). These responses are somewhat higher than the ones usually observed for sensors on alumina, which is ascribed to the lower elastic modulus of steel. The drift @ nominal pressure is very low up to 400 MPa for both systems. Nevertheless the results are more scattered with the low temperature system. Some of the scattering could be due to the effect of tightening the sensors, which was found to be of the order of 1%.

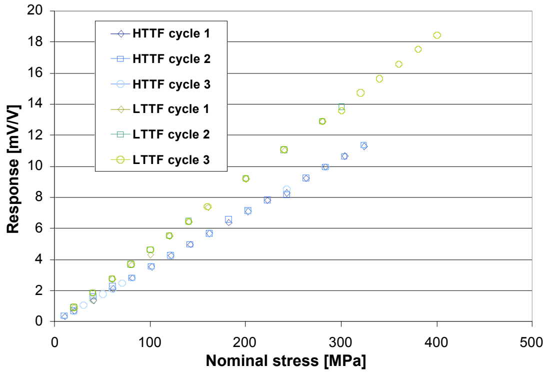

Figure 4: Response of HT sensors with the commercial high-firing TF system (HTTF) and the low firng TF system (LTTF) for 3 cycles

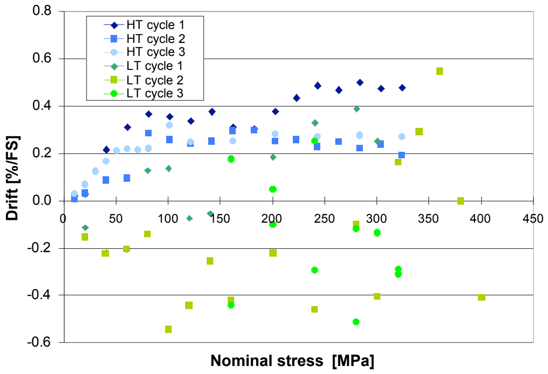

Figure 5: Drift of HT sensors with the commercial high-firing TF system (HTTF) and the low-firing TF system (LTTF) for 3 cycles

TF on 17-4 PH steel

This section presents the results of the 17-4 PH membranes (machined and MIM) screen-printed with the low temperature TF system and compared to the HT steel membranes with the high temperature TF system as reference. With the low temperature TF system, the span @ nominal pressure is 5.64 ± 0.02 mV/V for the 17-4 PH sensor vs. 4.94 ± 0.01 mV/V for the HT steel (Figure 6). Figure 7 presents the drift after 4 cycles (140, 150, 200 and 160 bar) of 17-4 PH sensors compared to HT steel sensor which has only been loaded 3 times at 160 bar. The drift of the 17-4 PH sensors are very low (<1% of the span) but more scattered than that of the HT sensor.

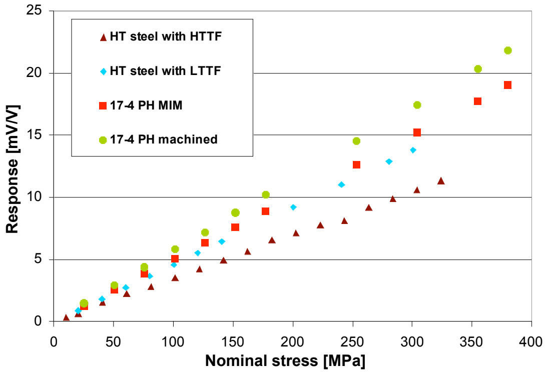

Figure 6: Response of 17-4 PH sensors compared to the HT sensors with the HTTF system and the LTTF system for the second load cycle

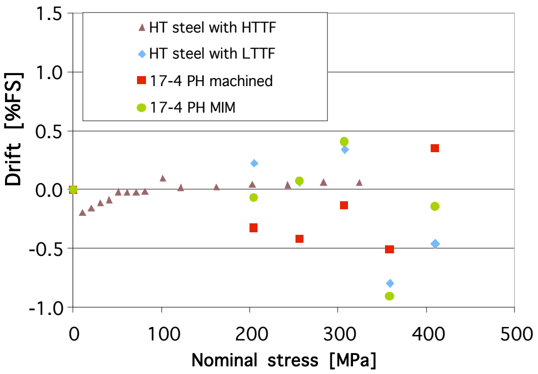

Figure 7: Drift of 17-4 PH sensors after 4 load cycles compared to the HT sensors with the HTTF system and the LTTF system

TF on A 286 steel

Figure 8 presents the response of the A286 sensor compared to the HT sensor, with the HTTF system and the LTTF system. The span of A286 sensor is 4.75 ± 0.06 mV/V, quite similar to the HT sensor with the LTTF system. Figure 9 depicts the drift of the A286 sensor after 4 load cycles (140, 150, 200 and 160 bar). As for the 17-4 PH, the drift is very low but more scattered compared to the HT sensor with the HTTF system.

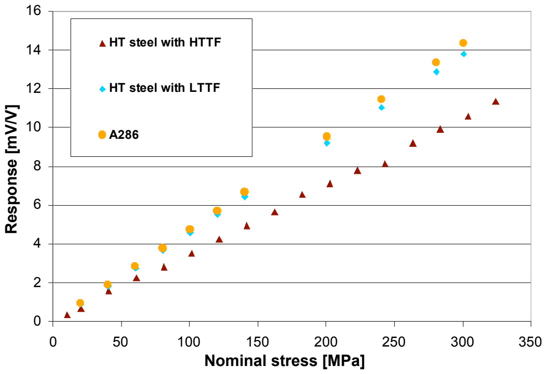

Figure 8: Response of the A286 sensor compared to the HT sensors with the HTTF system and the LTTF system for the second load cycle

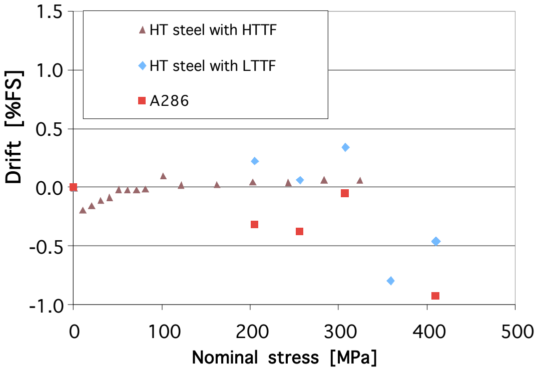

Figure 9: Drift of A286 sensors after 4 load cycles compared to the HT sensors with the HTTF system and the LTTF system

Force sensor used in surgical operation of total knee arthroplasty (TKA)

The austenitic steel A286 being chemically similar to the medical alloy, the same TF system was then used for a ligament balancing sensor for the total knee arthroplasty operation (Figure 10).

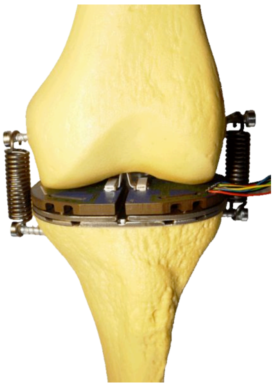

Figure 10: force sensor in bone setting

The Figure 11 depicts the sensor composed of 2 parts on which 3 force sensing bridges by parts have been deposited.

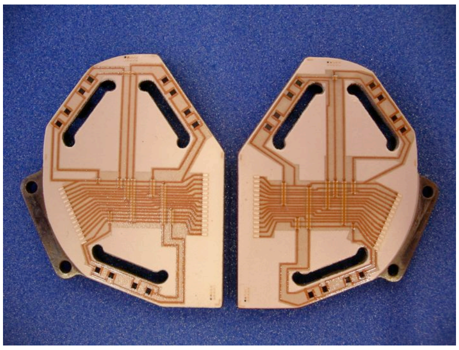

Figure 11: ligament balancing sensor for total knee arthroplasty

4. Conclusion

The goal of this work was the characterization of steel sensors based on low fired thick-film technology previously developed. A high-performance pressure sensor based on several high-strength steel substrates, chemically similar to the ferritic and austenitic steels was principally used for this characterisation. Several steels have been used to evaluate the TF performance. We have obtained reliable systems for ferritic and austenitic steels with high resistance to stress. Nevertheless, if the drifts are very low, they are negative, which induce that it is due to the LTTF system. Moreover the drifts are more scattered than those obtained with the commercial TF system. The LTTF systems have still to be optimised.

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