High performance thick-film pressure sensors on steel

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Abstract

The aim of this work is to examine the possibility of high performance pressure sensors based on piezoresistive thick films deposited on steel membranes, which combine the performance advantage of thin-film sensors (high-strength steel substrates, assembly without elastomer seal) with the low cost of ceramic thick-film sensors. As standard thick-film firing conditions degrade the properties of most high-strength steels, two routes were explored: 1) application of a special steel, which does not undergo mechanical degradation at 850°C, and 2) development and use of thick-film materials firing at a lower temperature.

This work presents the development and characterisation of low-firing thick-film systems (dielectrics, resistors and conductors), formulated to achieve chemical and thermal expansion compatibility with a wide range of substrates. Results on electrical properties of these systems: resistance, thermal coefficient of resistance (TCR) and strain response on different steel substrates are compared, together with those of standard thick-film systems on heat resistant special steel.

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

1 Introduction

Pressure sensors based on thick-film 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 [1]. Additionally, the applications of this kind of sensors remain limited because their assembly requires elastomer seals. Stainless steels [2] 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-strength steel, owing to degradation of mechanical properties due to annealing or dimensional changes associated with martensitic transformation (which tend to destroy the 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. 600°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 this work, we present the results of these two routes, i.e. 1) piezoresistive sensors using commercial 850°C-firing thick films on special high-temperature steels, and 2) sensors based on a low-temperature thick-film system on common precipitation hardening martensitic stainless steel. For this last solution, we also study and discuss the electrical results from test patterns compared with standard alumina-based systems: sheet resistance (SR), thermal coefficient of resistance (TCR) and gauge factors (GF).

2 Experiments

2.1 Substrates

For the sensors, the following substrate materials were used: a) a special high-temperature resisting steel, denoted HT, b) Special Metals A286, and c) 17-4 PH / EN 1.4542. 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. A286 and 17-4 PH, on the other hand, require a lower temperature thick-film process, and are compatible with maximum firing temperatures of ca. 750°C and 650°C respectively. 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. 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 sheet (AK Steels) for electrical and piezoresistive characterisation, and 2) parts by metallic injection molding (MIM, Figure 1), made with two sintering temperatures (1340°C and 1350°C). Finally, 96% pure alumina (Kyocera, Japan, A-476, TCE = 7 ppm/K) has been used as a standard reference substrate.

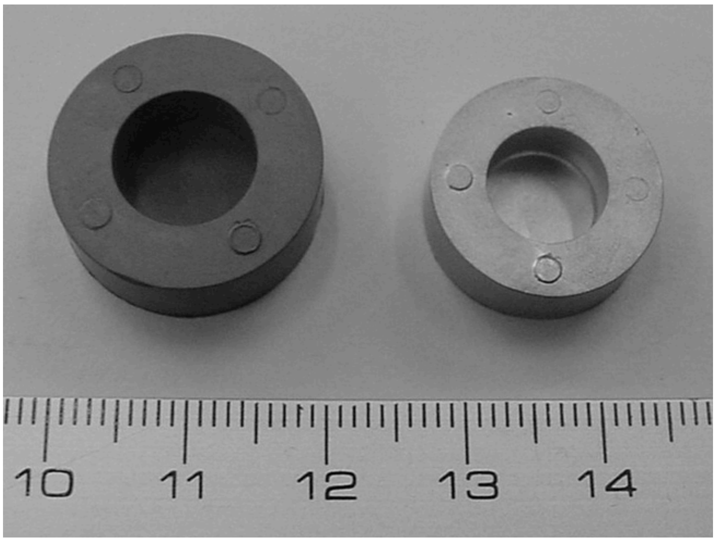

Figure 1: MIM green and sintered 17-4 PH cells

2.2 Thick-film systems

Overall, 4 thick-film systems, listed in Table 2, were applied: one classical, 850°C firing and using only commercial materials, and 3 low-temperature systems. Each thick-film system comprised 3 functional layers, deposited in the following sequence: 1) the dielectric, with 3 screen-printed and separately fired layers (total thickness: ca. 75 μm) in order to guarantee good insulation, 2) the conductors / resistor terminations, and 3) the resistive composition.

The commercial system 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).

Three low-firing thick-film systems have been developed, aiming to match the thermal expansion coefficient of metallic substrates and to achieve optimal properties while lowering the firing temperature. First, in order to guarantee the insulation, three dielectrics, based on 2 lead borosilicate glasses (denoted V6 and V8), loaded with different fillers, were produced and are listed in Table 1. The filler powder (quartz or cristobalite) serves to dimensionally stabilise the dielectric, and to control its TCE. The average particle size is around 3 μm for quartz powder and around 2.5 μm for cristobalite powder.

Table 1: List of the studied dielectric compositions.

Code	Glass variant	Filler
V6C55	V6	55% vol cristobalite
V6Q55	V6	55% vol quartz
V8Q40	V8	40% vol quartz

The composition (by mass) of the glasses is V6: 75% PbO + 10% B_2O_3 + 15% SiO_2, and V8: 85% PbO + 10% B_2O_3 + 5% SiO_2. In both cases, 2% Al_2O_3 was added to inhibit crystallisation [3]. The ideal firing temperature is around 600°C for V6-based materials and around 500°C for V8-based ones.

For the 600°C range, we used commercial ESL 9912 Ag conductor and ESL 3114 resistor (10 kOhm, 625°C firing for porcelain enamelled steel). Finally, for the low temperature 500°C range, we used a 3:1 (vol.) mix of ESL 9912 with V8 (used as a frit), and an experimental resistive composition consisting of V8 glass loaded with RuO_2 nano-powder.

For low-firing compositions, the firing cycle started with a 25 min dwell at 370°C for organic burnout, followed by a 15 min dwell time at the firing temperature. The standard materials used a simple firing cycle, with a 10 min dwell time at 850°C.

Table 2: List of sample series and firing temperatures

Substrates	Dielectrics	Conductor	Resistor
HT	Commercial 850°C	ESL9635B 850°C	Du2041 850°C
A286	V6C55 625°C	ESL9912 600°C	ESL3114 625°C
17-4-PH	V6Q55 625°C	ESL9912 600°C	ESL3114 625°C
17-4-PH	V8Q40 525°C	ESL9912 : V8 500°C	V8 resistor 525°C

2.3 Samples

Test samples for sheet resistance (SR) and TCR were 1.5 mm wide resistors of several lengths (Figure 2). Sheet resistance and TCR were measured as a function of resistor length at 30°C and 100°C. GF was determined at 30°C by applying appropriate loads at the end of test cantilevers (Figure 3). Test samples deposited on the alumina reference substrate only comprised the conductor and resistor, as the dielectric is not thermally matched to alumina.

The integrated metallic pressure sensor, depicted on Figure 4, is composed of a membrane (diameter 9.3 mm, thickness ca. 0.8 mm), on which a thick-film piezoresistive bridge is deposited. The nominal pressure, for ~100 MPa stress, is 40 bar. The cells have been measured with a hydraulic dead weight pressure tester DH-Budenberg 540 VHX. 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.

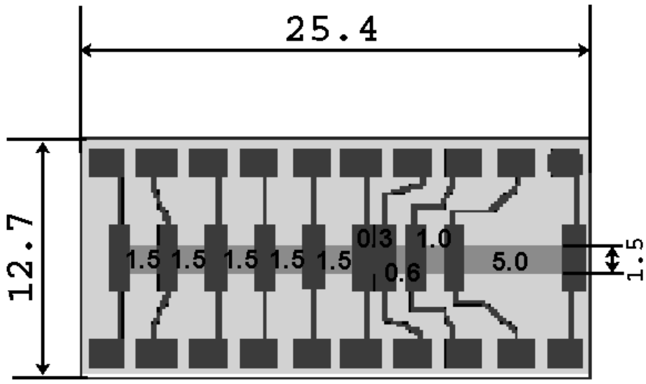

Figure 2: Layout of the test samples for SR and TCR measurements

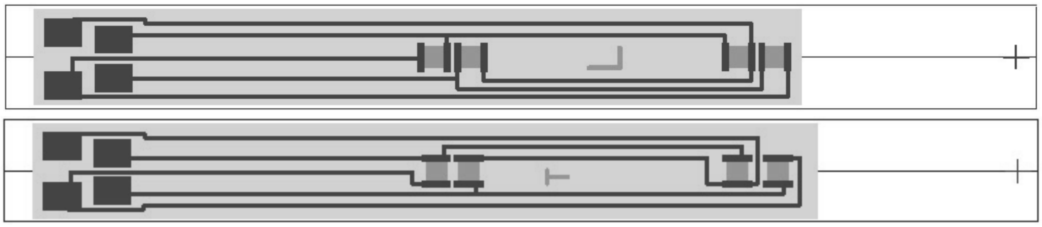

Figure 3: Layout of samples for piezoresistive measurement (depicted here: longitudinal and transverse)

Figure 4: MIM sintered parts (top and bottom)

3 Sheet resistance and TCR measurements of low temperature thick-film systems

Table 3 presents the sheet resistance, the TCR and the longitudinal and transverse GF values of resistors on 17-4 PH steel substrates. Globally, the measured properties are very reproducible, especially TCR and GF. Sheet resistance tends to be higher on steel+dielectric than on alumina, which suggest that part of the resistor thickness is “lost” by interaction with the dielectric. While GF values on steel+dielectric and on alumina are similar, a strong TCR shift is observed, which can be ascribed to thermomechanical and chemical effects [4].

Overall, the properties of the semi-custom 625°C-firing system are excellent on 17-4 PH steel. On the other hand, the 525°C-firing system shows mixed results. The resistor formulation should be modified to lower TCR. Moreover, the very high gauge factor is not necessarily, an advantage, as this is often linked with higher noise and poorer stability.

Table 3: Electrical results of thick-film resistors

Dielectric	V6Q55	V8Q40
Conductor	ESL 9912A	ESL 9912A:V8
Resistor	ESL 3114	V8 resistor
Res. firing temperature	625°C	525°C
Sheet resistance (kΩ)	21 ± 2 (Al_2O_3: 11 ± 1)	50 ± 10 (Al_2O_3: 19 ± 5)
TCR (ppm/K)	13 ± 6 (Al_2O_3:-220 ± 15)	578 ± 7 (Al_2O_3: 363 ± 8)
Gauge Factor (longitudinal & transverse)	L: 12.2 ± 0.2 T: 9.4 ± 0.1 Al_2O_3: (L)10.3 ± 0.2 (T)7.7 ± 0.2	L:23.5 ± 0.2 T:18.9 ± 0.2 Al_2O_3: (L)23.0 ± 0.3 (T) 19.1 ± 0.2

4 Performance of cells under pressure

Each cell have undergone 3 loading cycles, by repeatedly loading / unloading the samples with increasing pressures up to a maximum pressure of 160 bar. The drift is presented for each cycle relative to the zero-load value after loading at 40 bar.

4.1 HT, screen-printed with commercial thick-film system fired at 850°C

The properties of this combination are excellent. The signal of this cell is stable: the span is 2.839 ± 0.007 mV/V at the nominal pressure 40 bar. The drift of the cell is 0.06 % at 160 bar (400 MPa), and adhesion of the thick-film system is good (Figure 5).

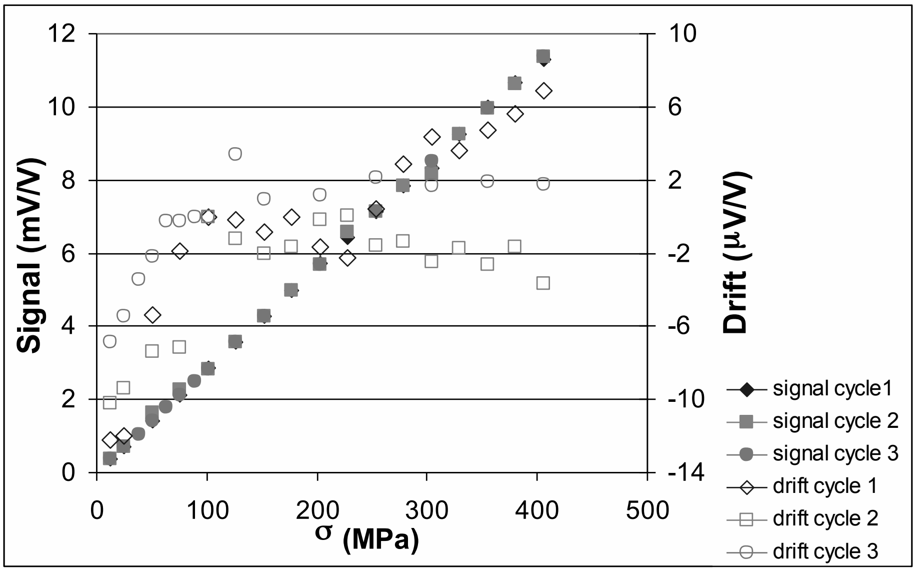

Figure 5: Signal and drift of "HT" sensor

4.2 A286, screen-printed with mixed developed and commercial thick-film system fired at 625°C

The span of this cell is 1.83 ± 0.05 mV/V at the nominal pressure 40 bar. Its drift is 6.9 % at 160 bar (400 MPa, Figure 6).

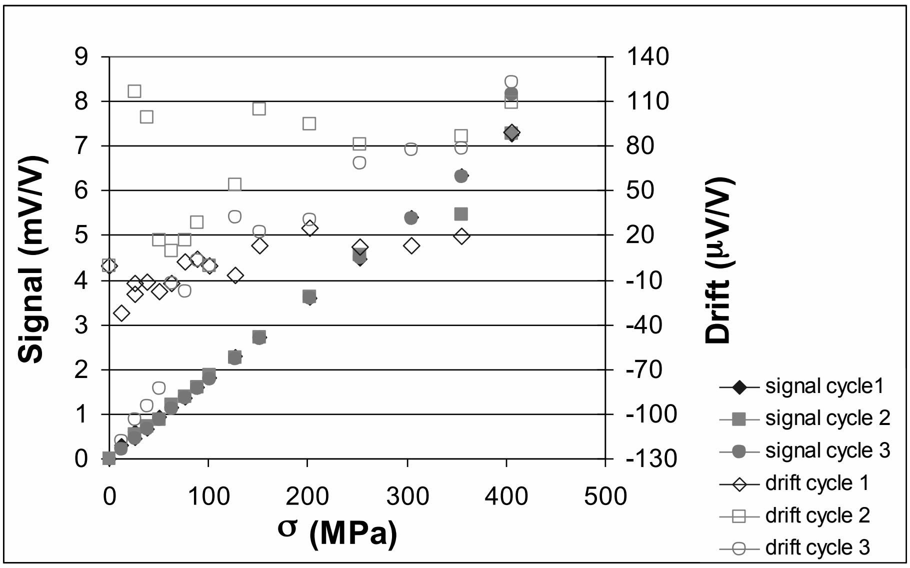

Figure 6: Signal drift of A286 sensor

4.3 17-4 PH cells screen-printed with mixed developed and commercial thick-film system fired at 625°C obtained by MIM and sintered at 1340°C and 1350°C

The span of the cell sintered at 1340°C is 3.51 ± 0.02 mV/V at the nominal pressure 40 bar. Its drift is 4.2 % at 160 bar (400 MPa, Figure 7).

The span of the cell sintered at 1350°C is 2.37 ± 0.02 mV/V at the nominal pressure 40 bar. The drift of the cell is 1.7 % at 140 bar (~350 MPa, Figure 8).

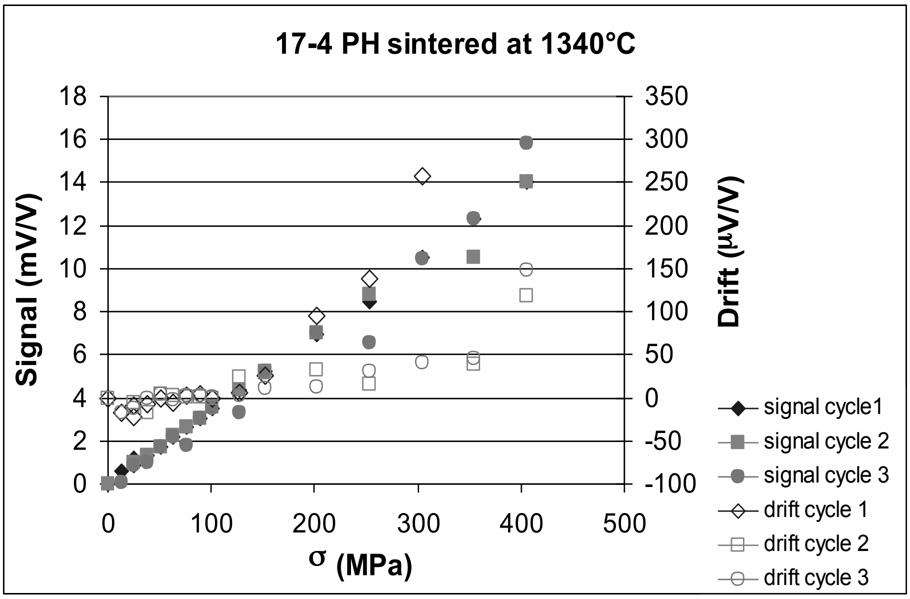

Figure 7: Signal and drift of 17-4 PH cell sintered at 1340°C with 625°C thick-film process

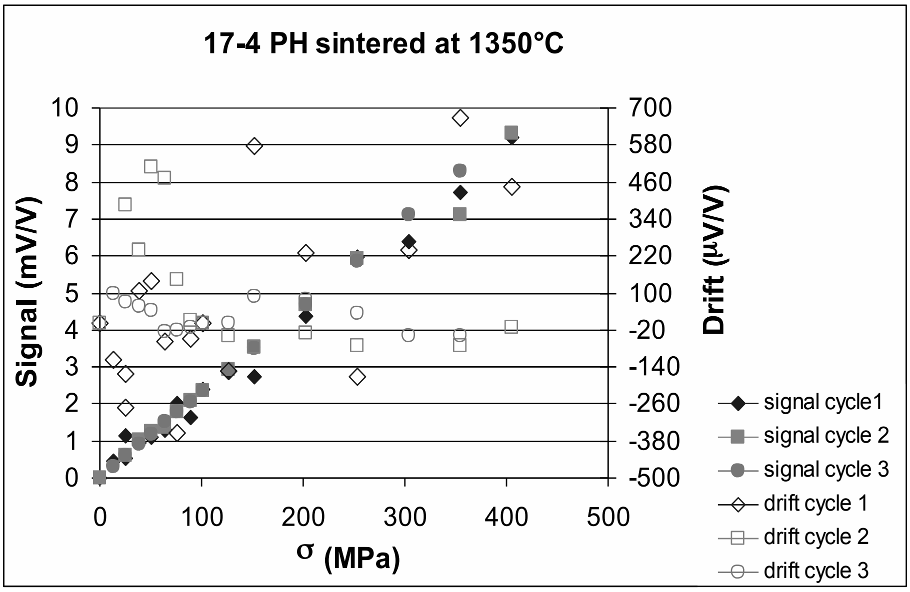

Figure 8: Signal and drift of 17-4 PH sintered at 1350°C with 625°C thick-film process

4.4 *17-4 PH cells screen-printed with low temperature thick-film system fired at 525°C obtained by MIM sintered at 1340°C and 1350°C*

Loss of the signal is experienced at rather low pressure, namely 120 bar (300 MPa), and the drift increases from ~50 bar (~130 MPa) on. This is ascribed to insufficient adherence of our 525°C-firing system. The span of the cell sintered at 1340°C is 3.2 ± 0.3 mV/V at the nominal pressure 40 bar. The results are depicted on Figure 9.

For the sensor sintered at 1350°C (Figure 10), it is not possible to give the span because of the high dispersion of results due to the damage on the thick-film bridge.

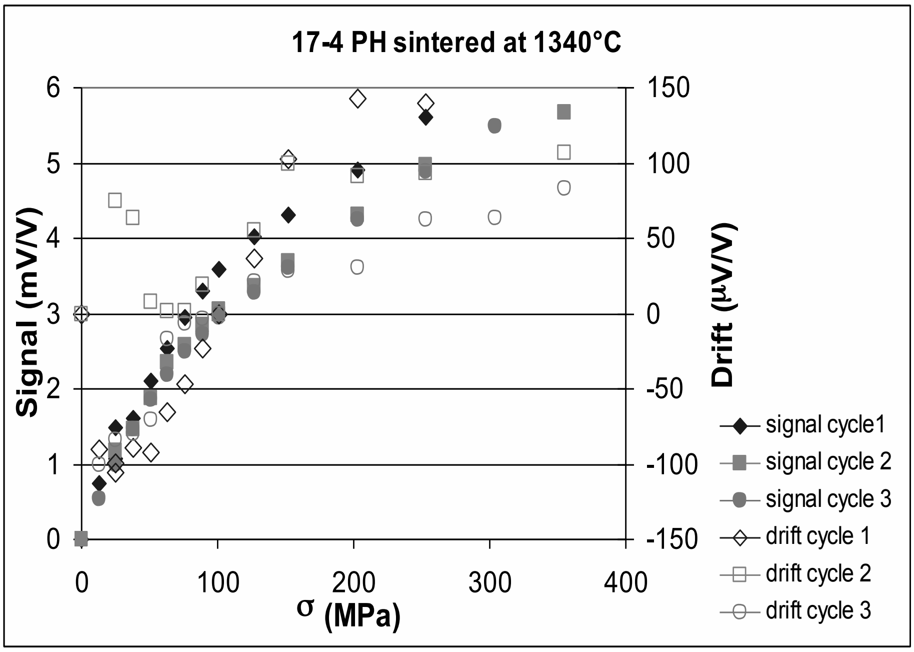

Figure 9: Signal and drift of 17-4 PH sintered at 1340°C with 525°C thick-film process

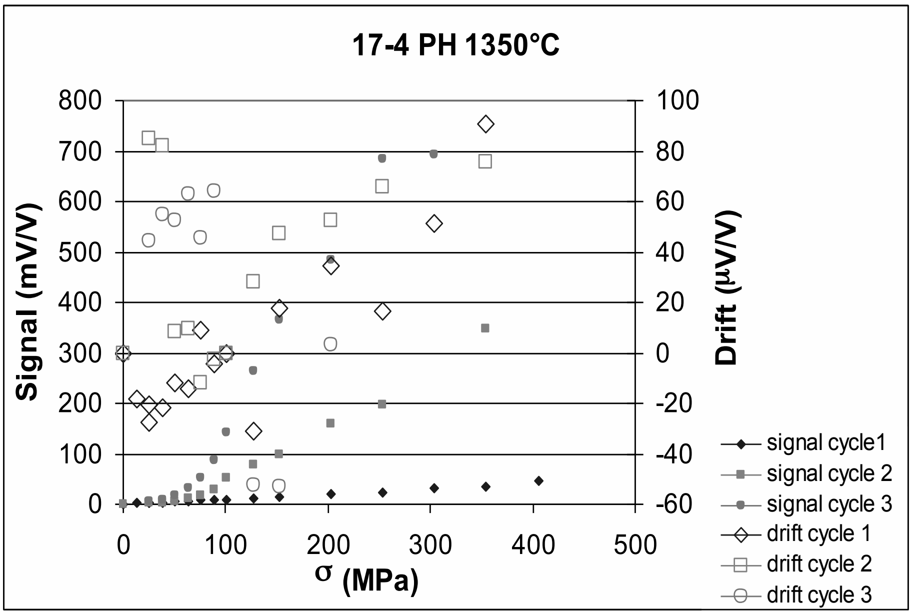

Figure 10: Signal and drift of 17-4 PH sintered at 1340°C with 525°C thick-film process

We must note that some problems may be due to poor contacts of the cables to the pressure cells using low-firing systems, due to some diffusion of the dielectric glass into the conductors.

Figure 11 represents the comparison of drift of zero (%) during the third cycle between 0 and 400 bar. The drift of the sensors with low-firing thick-film systems is not typical of plastic deformation of the metallic substrate, which would result in a progressive increase of drift with pressure [1]. Our more scattered results, especially for the 525°C-firing system, rather indicate that the thick-film bridge experiences damage such as cracks and loss of adhesion. In fact, loss of adhesion was experienced in a few extreme cases, which was not unexpected as our model dielectrics did not yet comprise adhesion promoters such as Co or Ni.

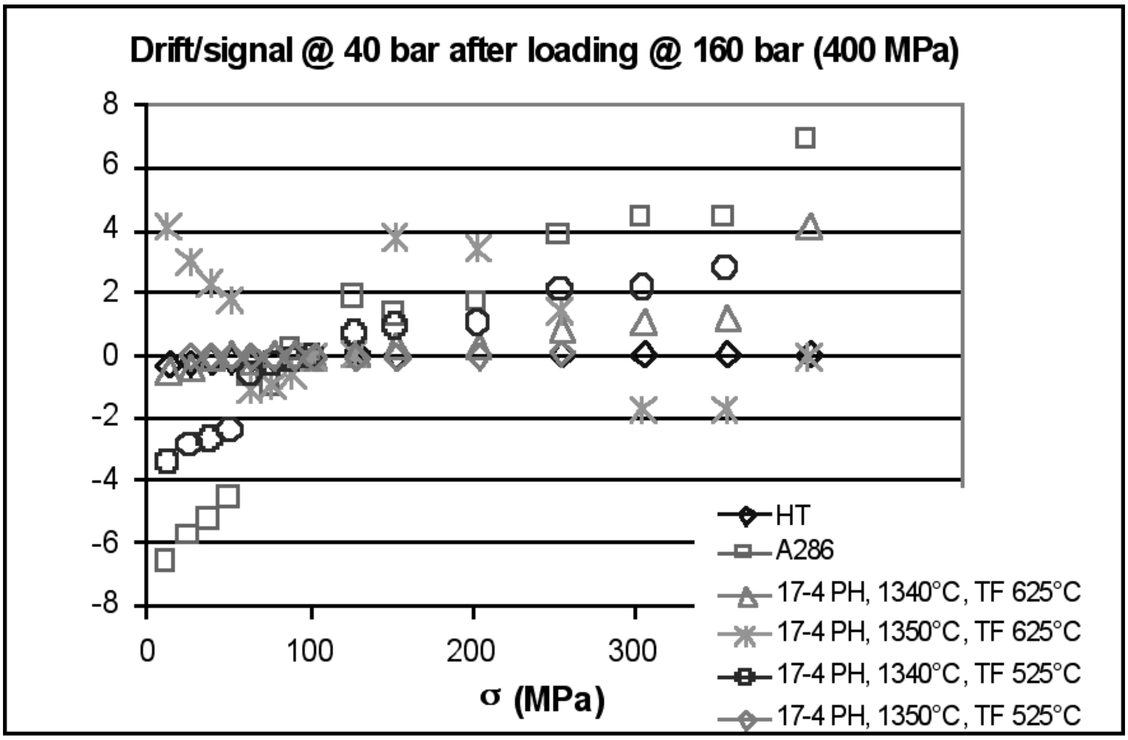

Figure 11: Drift/signal @ 40 bar after loading @ 160 bar (400 MPa)

5 Conclusions and outlook

The study has characterised several steel / thick-film combinations in an attempt to optimise thick-film piezoresistive sensors on steel substrates. A special steel grade ("HT") was found, which allows to use commercial materials with excellent results. Low-temperature thick-film systems were also developed in order to achieve compatibility with more common steel grades. Especially, excellent electric and piezoresistive properties were achieved for a 625°C-firing system. However, stability under high loads still must be improved, which will be attempted by optimising the dielectrics, especially by incorporating adhesion promoters.

6 Acknowledgements

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7 References

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