High-strain response of piezoresistive thick-film resistors on titanium alloy substrates

C. Jacq, Th. Maeder, P. Ryser

Laboratoire de Production Microtechnique, Ecole Polytechnique Fédérale de Lausanne, Station 17, CH-1015 Lausanne, Switzerland.

Version of record: Journal of the European Ceramic Society 24 (6), 1897-1900, 2004.
http://hdl.handle.net/10.1016/S0955-2219(03)00467-9

Abstract

We examine in this work the integration and high-strain response of piezoresistive thick-film resistors on titanium and titanium alloy, as compared to ferritic stainless steel and the standard alumina substrates. In general, titanium and its alloys are relatively ill suited for use as a thick-film substrate using the standard 850 C firing process, because of its low oxidation resistance and tendency to dissolve oxygen. This problem can be alleviated by applying suitable thick-film glassy protection layers. Titanium alloys are very elastic, and allow piezoresistive responses well in excess of 4% with little plastic deformation of the substrate, using standard commercial thick-film resistor compositions. In this case, the behaviour of the sensor becomes asymmetric, as failure of the resistor or insulating dielectric under tension – and therefore the mechanical properties of these materials – becomes the factor limiting the response of the sensor.

Keywords: *films (A), mechanical properties (C), plasticity (C), sensors (E), substrates (E)*

1 Introduction

Thick-film technology used for piezoresistive force or pressure sensing typically uses alumina as a substrate material[1] because it is the standard for thick-film technology. But alumina is not optimal for piezoresistive sensing applications, as its elastic modulus is high and its strength rather low. For applications, design stress is limited at ca. 100 MPa. Piezoresistive response could be considerably increased by using a more flexible substrate. Recently[2, 3], sensors based on stainless steel have been introduced. Steels offer advantages such as robustness and ease of fabrication. However, the high temperatures associated with thick-film processing (850 C) are usually not compatible with good mechanical properties of the steels. Steels soften at high temperatures, and the dimensional changes associated with martensitic transformation tend to destroy the thick-film layers. Titanium alloys have low elastic modulus and potentially very high strength, as the solution annealing temperatures of beta titanium alloys are compatible with those used in firing of thick-film layers. We therefore expect to reach very high elastic strains, and hence piezoresistive responses. In this case, response may be limited by mechanical failure of the thick-film layers rather than by the substrate. Titanium and its alloys however tend to oxidise rapidly at high temperatures in air, and must be suitably protected. In this work, we endeavour to investigate titanium and titanium alloys as elastic substrate materials for thick-film piezoresistive sensors, compared with stainless steel and alumina. Relevant parameters such as drift, gauge factor and maximum response are studied and discussed.

2 Experimental

The test device is a cantilever beam with four thick-film strain-sensitive resistors configured in a Wheatstone bridge arrangement. A weight F applied to the free end of the beam causes a surface strain to occur in the elastic beam. The magnitude of the strain is a function of the weight, the substrate material and the beam geometry. The electrical layout of the thick-film measurement cell is depicted in Fig. 1. The resistors on the left side of the beam experience a more important strain than those on the right side. Two variants exist, for longitudinal and transverse piezoresistive characterisation.

In this work, the beams (substrates) were made from scored substrates (alumina) and from laser cut sheet metal. 5 materials were used. 96% pure alumina (Kyocera, Japan, A-476, “alumina”) is the standard substrate material used for thick-film circuits. Steel 1.4016 (Ugine, France, “1.4016”) is a Fe-17% Cr ferritic stainless steel where solid solution is the only strengthening mechanism. T40 (ACNIS, France, “T40”) is commercial grade 2 unalloyed titanium. Finally the titanium alloys Timetal 21s and Timetal 15-3 (Timet Corp., USA, “Ti 21s” and “Ti 15-3”) are metastable high-strength beta titanium alloys.

The geometric data for the beam, materials properties and insulating dielectric composition for the 5 substrate materials are listed in Table 1. For Ti 15-3 and Ti 21s, the elastic moduli are approximate, as they are processing dependent. The effective modulus (for plane strain) has been calculated by the following expression.

$$E^* = \frac{E}{1-\nu^2} \qquad (1)$$

E	Young's elastic modulus
E^*	Effective elastic modulus
ν	Poisson's coefficient

The thick-film Wheatstone bridge was applied with a succession of standard screen-printing, drying and firing cycles. The dielectric (Table 1) was applied as an insulating medium, followed by the conductor (ESL 9635B, Ag:Pd 3:1) and resistor (Du Pont 2041, 10 kOhm nominal value) compositions. Each layer is fired separately at 850 C during 10 minutes. Titanium and its alloys were found to require a glassy protection layer prior to deposition of the proper dielectric in order to ensure adhesion to high elastic strain.

The beams, clamped at one end, were tested by successively applying and removing increasing bending moments. Signal and drift are measured after unloading for each point. All materials were initially tested with the thick-film bridge on the bottom (compression). Metallic beams were also tested with the bridge on the top (tension). 1.4016 steel beams were found to be very plastic after thick-film processing, and were therefore subjected to a preloading treatment at 400 MPa in order to improve their stability.

From beam theory, for a bending beam, the maximal stress for a given load is given by:

$$\sigma = \frac{6.l}{b.h^2} \cdot F \qquad (2)$$

σ	maximal stress (nominal)
F	applied load
b	width of the sample
l	effective length, between assembly point and load point
h	thickness of sample

This value is nominal and does not account for mounting and geometric imperfections.

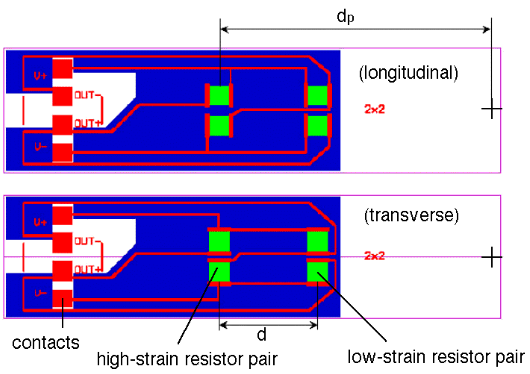

Figure 1. Layout of the test cantilever beams..

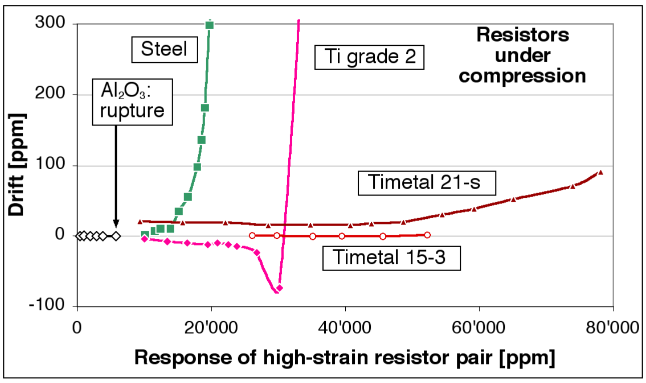

Figure 2. Drift as a function of piezoresistive response of the most stressed resistors.

Material	Alumina	**1.4016**	T40	Ti 21s	Ti 15-3
Elastic modulus *E* (GPa)	315	210	120	100	100
Poisson coeff. ν	0,23	0,27	0,3	0,3	0,3
Effective modulus *E** (GPa)	333	227	132	110	110
Total length (mm)	50.8	50.8	50.8	50.8	50.8
Width *b* (mm)	12.7	12.7	12.7	12.7	12.7
Thickness *h* (mm)	0.5	0.5	1	0.8	0.5
Dielectric	Heraeus GPA98-029 or ESL 4702	Heraeus GPA 98-029	ESL 4702*	ESL 4702*	ESL 4702*

Table 1: Materials and geometric parameters for the cantilever beams.
*** Onto glassy protection layer.**

3 Results and discussion

3.1 Comparison of substrate materials

In order to compare the substrate materials, the samples were first tested with the thick-film measurement bridge under compression. This is necessary in order to avoid being limited by mechanical failure of the glassy, brittle dielectric and resistor layers, which are expected to be much stronger in compression than in tension. Fig. 2 gives the drift of the signal as a function of the response (absolute value) of the most stressed pair of resistors, for samples of the longitudinal variant (electric current parallel to strain direction). The maximum responses, strain and stresses before failure are given in Table 2, together with the longitudinal (GF_L) and transverse (GF_T) gauge factors. The response ΔR/R of the high-strain resistors has been calculated with the following expression.

$$\frac{\Delta R}{R} = \Delta signal \cdot 2\frac{d_p}{d} \quad (3)$$

These results show the considerably higher responses attainable with titanium alloys compared the other materials. For alumina, no drift is observed but failure occurs at low strain (ca. 1’500 ppm, 500 MPa stress). In practical applications, allowing for subcritical crack growth, statistical dispersion of strength and safety factors, strain and stress are limited at ca. 300 ppm / 100 MPa.

For 1.4016 steel (preloaded at 400 MPa, which is about maximum for this alloy), drift of the output signal is observed above this preloading stress (strain: 1’800 ppm). In this case, drift is clearly due to a (quite visible) plastic deformation of the substrate. Better results are observed for T40 titanium. Maximum stress is not much higher (500 MPa), but the lower elastic modulus allows for considerable improvement in strain (3’800 ppm).

Material	GF_L	GF_T	Max. strain (ppm)	Max. stress (MPa)	ΔR/R (ppm)
Alumina	12.3	7.7	1’500	530	5’700
1.4016	9.9	6.4	1’800	450	19’500
T40	10.1	5.3	3’800	500	26’800
Ti 21s	10.6	6.4	6’600	730	55’000
Ti 15-3	9.0	5.2	7’300	≥800	≥52’000

Table 2. Gauge factors, max. strain, stress and resistor response as a function of substrate material.

Very little drift is observed up to very high strains for the beta titanium alloys. In fact, Ti 15-3 samples couldn't be measured completely due to excessive elastic bending of the sample. Ti 21s presents a slight drift above 6'600 ppm / 730 MPa, which corresponds to 5% piezoresistive response. For this material, the slight drift observed at low stress is probably thermal in nature. As both titanium alloys are expected to be in the solution annealed state after thick-film deposition, these very high properties could probably still be improved by a subsequent aging treatment.

Values of the gauge factor are quite usual for the applied Du Pont 2041 composition[4]. The observed decrease on the metallic alloys vs. alumina is not due to direct resistor-dielectric interaction, as this effect was absent on alumina beams coated with the same dielectrics. The most probable cause is chemical interaction with the substrate through the rather thin (30 µm) dielectric layer. In the case of the titanium alloys, part of the observed decrease could be an artefact due to an oxidation-induced change of the alloy mechanical properties. Finally, another possible source of error is stiffening by transverse bending of the beams due to internal stresses. This problem will be addressed in future work by reducing the width of the cantilevers.

3.2 Limiting strain of the measurement bridge under tension

A second set of samples were tested with the measurement bridge under tension, in order to determine whether failure of the thick-film structure can become a limiting factor at high strain. Moreover, in this second series of measurements, beams with longitudinal and transverse resistors were compared in order to distinguish if the orientation of the resistor terminations have an influence on the failure strain.

The drift of the output signal and the gauge factor are given in Figs. 3 and 4, as a function the strain of the most stressed pair of resistors. Both values are found to be necessary, because the signal in some samples which failed under load was found to return to normal upon unloading. A summary of the results, compared with those under compression, is given in Table 3.

For 1.4016 steel (and alumina), premature failure of the measurement bridge is never observed. Failure of the substrate by plastic deformation (steel) or brittle fracture (alumina) is the limiting factor. In T40 titanium, failure of the bridge under tension is observed, but at strain levels somewhat higher than those necessary to cause plastic deformation of the substrate. A different situation arises with Ti 15-3 and Ti 21s: failure of the thick-film circuit in tension is observed at strain levels below those necessary to initiate plastic deformation of the substrate (see prior part). This is expected due to the very high elastic strains attainable with these alloys. From our limited number of samples, no reliable dependence of failure strain of the thick-film circuit on resistor orientation (transverse or longitudinal) could be observed.

Interestingly, failure of the thick-film circuit cannot be evidenced from drift measurements alone, as some samples where the electrical signal became aberrant under load actually exhibited little or no drift upon unloading. This calls into question our initial assumption that failure of the thick-film bridge under tension occurs in the measuring resistors. The fact that little drift is sometimes observed upon unloading rather seems to imply that failure also may occur in the dielectric near the fastening point of the cantilever, where the strain is highest ("substrate strain" in Table 3). In this case, a crack in the dielectric could break a conductor line. This electrical connexion would be closed again upon unloading, which would explain the absence of noticeable drift.

4 Conclusion

Titanium alloys are a very promising material for thick-film piezoresistive force or pressure sensing, provided the remaining issue of excessive oxidation of the uncoated surfaces is resolved. Their high strength due to compatibility of alloy thermal treatment with thick-film processing, coupled with a low elastic modulus, allow very high elastic strains and hence piezoresistive responses in excess of 5% with the measuring bridge under compression. Under tension, response is limited by mechanical failure of the thick-film circuit.

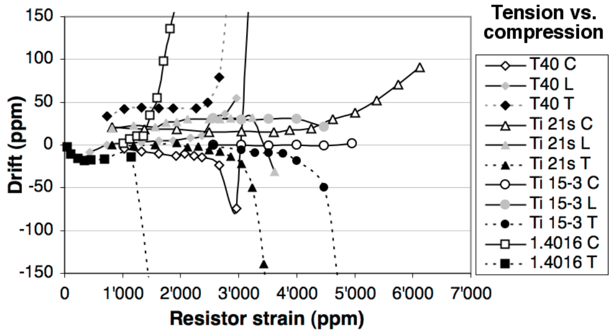

Figure 3. Drift as a function of strain (of the high-strain resistors). C = compression, longitudinal. L = tension, longitudinal. T = tension, transverse.

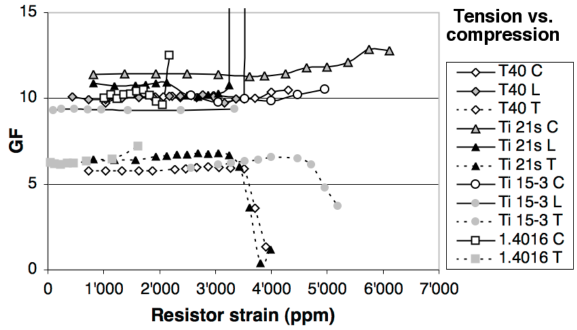

Figure 4. Gauge factor as a function of strain (of the high-strain resistors). Legends see Fig. 3.

Material	Compression Max substrate strain (ppm)	Tension Max substrate strain (ppm)	Tension Max resistor strain (ppm)	Tension Limiting factor
Alumina L	1’500	-	-	S
1.4016 (L & T)	1’800	1’800	1’250	S
T40 L	3’800	4’300	3’000	S
T40 T	-	3’900	2’700	S
Ti 21s L	6’600	4’600	3’200	TF
Ti 21s T	-	4’300	3’050	TF
Ti 15-3 L	≥7’300	≥6’500	≥4’500	?
Ti 15-3 T	-	5’800	4’000	TF

Table 3. Strain to failure, by excessive drift or rupture, in compression and tension, for each material. L = longitudinal (compression & tension) and T = transverse resistors (tension only). S = substrate, TF = thick-film.

References

1 White, N.M., A study of the piezoresistive effect in thick-film resistors and its application to load transduction, doctoral thesis, University of Southampton, Faculty of Engineering & applied Science, 1988.

2 Fraigi, L, Lupi, D. and Malatto, L., A thick-film pressure for cars propelled by natural gas, Sensors and Actuators A, 1994, 41-42, 439-441.

3 White, N.M. and Brignell, J.E., A planar thick-film load cell, Sensors and Actuators A, 1991, 25-27, 313-319.

4 Hrovat, M., Belavic, D. and Holc., J., Evaluation of some thick-film materials for temperature, force, and humidity sensors, MCM/C Mixed Technologies and Thick Film Sensors, 1994, 3:2 , 267-272.