Platinum Alloy Strain Gauge Materials

NOBLE METAL ALLOYS FOR STATIC STRAIN MEASUREMENT AT 900°C

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For over 30 years there has been research into materials that could be used for strain gauges operating over the temperature range 700 to 1200°C. At these temperatures strain gauges have a number of uses, for example, they can measure the stress distribution and fatigue life of turbine blades in rapidly rotating turbojet engines and the high temperature instantaneous hot stress of the helm structure and jet nozzle; they can be used in research for the design codes of new types of aircraft, and for failure analysis.

A number of commercially available high temperature strain gauges, some using the platinum group metals, have been developed. The platinum group metal materials used in these strain gauges are, in weight per cent: platinum-(8.5~9.5)tungsten (Pt-(8.5~9.5)W) (1), platinum-8 nickel-2 tungsten (Pt-8Ni-2W), platinum-8 nickel-2 chromium (Pt-8Ni-2Cr) (2), and palladium-13 chromium (Pd-13Cr) (3). Strain gauges containing non-noble metals are: copper-nickel (Cu-Ni), nickel-chromium (Ni-Cr) and iron-chromium-aluminium (Fe-Cr-Al) (4).

A half-bridge strain gauge made of Pt-8W alloy, capable of measuring strain at 650°C was produced successfully by R. Bertodo of Bristol Siddeley Engines Ltd., while W. T. Bean extended the maximum usable temperature of the platinum-nickel system to 760°C and J.-F. Lei increased the maximum usable temperature of Pd-Cr alloy to 800°C.

In general, the perfect material for a wire strain gauge (which records the change in electrical resistance) should be an oxidation-resistant pure metal of high melting point. The noble metals, when pure and unalloyed, have a very high temperature coefficient of resistance (TCR) and their electrical resistance is sensitive to temperature changes, so, unalloyed, they are unsuit-able for wire resistance strain measurements.

Alloy systems of Cu-Ni, Ni-Cr and Fe-Cr-Al have variable and very low temperature coefficients of resistance (~ 0), and can be made into single wire temperature-self-compensating strain gauges. The Fe-Cr-Al system exhibits microstructural instability over the temperature range 400 to 600°C; this results in poor linearity for the electrical resistance/temperature relationship, unsatisfactory reproducibility of resistance at low temperatures, and large thermal hysteresis. During operation the zero point cannot be adjusted, so each strain gauge made from Fe-Cr-Al must only be used for a material structure which possesses a similar coefficient of linear expansion. These deficiencies have limited the use of Fe-Cr-Al strain gauges; therefore platinum group metal alloys were examined as strain gauge materials.

The addition of alloying elements to the platinum group metals improves their electrical properties and decreases their temperature sensitivity. Alloying elements are generally chosen according to the Hume-Rothery Rules, so as to form a perfect solid solution in which the atoms have similar atom radii, valence and electronegativity. The major problems for strain gauge materials above 700°C are:
- there are changes in the microstructure of the alloy caused by the high temperature
- properties are unstable, due to oxidation and evaporation of the alloy components
- the high temperature strength of the alloy.

Platinum-Tungsten Alloys

The Pt-(8.5~9.5)W alloy is a good strain gauge material for temperatures below 700°C. However, at temperatures over 700°C, the vigorous oxidation and evaporation of the tungsten makes the zero drift of the strain gauge unacceptable. Nevertheless, based on earlier

Alloy Composition, weight per cent							
Alloy	W	Re	Ni	Cr	Y	Ir	Pt
1	8.5	4	2	0.5	-	-	balance
2	9.8	3.2	0.55	1.3	0.15	-	balance
3	-	-	0.5	0.6	0.05	5	balance

Alloys 1 and 2 are the working wires; alloy 3 is the compensating wire

research (5–8), and on general standards for solid solutions, Pt-(8.5~9.5)W was selected as the base alloy for our high temperature strain gauge material.

Rhenium was chosen as an alloying element to improve the high temperature strength and to decrease the TCR of the Pt-W alloy, while the oxidation-resistant elements, nickel and chromium, of similar atomic radius to platinum, were used as alloying elements to decrease the oxidation and evaporation of the tungsten above 800°C. Yttrium was added to decrease the grain size, and improve the adhesions of the surface oxidation layer to the oxidation layer, and of the oxidation layer to the base alloy.

The resulting alloys formed were: platinum-tungsten-rhenium-nickel-chromium (Pt-W-Re-Ni-Cr) and platinum-tungsten-rhenium-nickel-chromium-yttrium (Pt-W-Re-Ni-Cr-Y) and the compensating alloy: platinum-iridium-nickel-chromium-yttrium (Pt-Ir-Ni-Cr-Y), all for use at 900°C.

The internal structure, physical and mechanical properties, and the oxidation mechanism of these alloys were studied by X-ray diffraction, transmission electron microscopy (TEM), scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS) and optimum alloy compositions were determined. The new alloy systems possess excellent high temperature oxidation resistance, low, stable temperature coefficients of resistance, and their maximum usable temperature was at least 1000°C.

Alloy Preparation and Properties

The nominal compositions for the working wires and the compensating wire used in this study are listed above in the Table. The alloys were then processed for use as strain gauge materials by treatment with the following stages: high frequency induction melting (in an argon atmosphere) → hot forging → hot rolling → hot drawing → cool drawing → semifinished product → preoxidation treatment → end product.

Electrical Properties

Changes in the electrical resistance of the alloy wires in air have been previously measured (5). At 700°C the Pt-9.5W alloy was known to possess excellent oxidation resistance, which could be maintained, almost unchanged, for over 120 minutes.
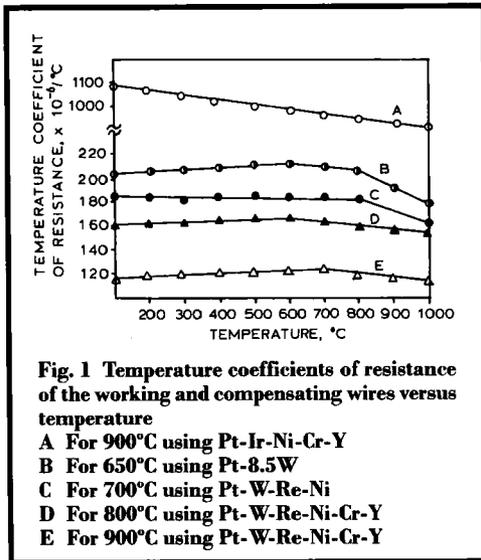
The relationships between the TCR and temperature (T) for several platinum alloys are shown in Figure 1. The TCR/T relationship for Pt-8.5W alloy, which is used to measure strain at 650°C, is a straight line over the temperature range 0 to 600°C; beyond 650°C linearity deteriorates.

For measurements at 800°C, using Pt-W-Re-Ni-Cr-Y alloy, the relationship from 0 to 600°C is a straight line and good linearity is maintained from 600 to 1000°C. Although the TCR of Pt-W-Re-Ni-Cr-Y alloy is smaller than that of Pt-W alloy, its absolute value is quite high (160×10^{-6} °C^{-1}).

For the newly developed Pt-W-Re-Ni-Cr-Y alloys for use at 900°C (with different components to the Pt-W-Re-Ni-Cr-Y alloy for use at 800°C) the linearities of their TCR/T relationships are good and the alloys possess much smaller TCR ($\alpha_{0-100} = 90$–120×10^{-6} °C^{-1}).

Phase Structure

Differential thermal analysis (DTA) shows that alloys containing additions of rhenium, nickel, chromium and yttrium remain as solid solutions. The DTA curve of this alloy is

Fig. 1 Temperature coefficients of resistance of the working and compensating wires versus temperature
A For 900°C using Pt-Ir-Ni-Cr-Y
B For 650°C using Pt-8.5W
C For 700°C using Pt-W-Re-Ni
D For 800°C using Pt-W-Re-Ni-Cr-Y
E For 900°C using Pt-W-Re-Ni-Cr-Y

similar to that of Pt-W. There is no phase transformation over the temperature range 500 to 1000°C.

Oxidation and Evaporation

The weight gains of four Pt-W alloy systems were measured under oxidising conditions at temperatures of 800 and 900°C by thermogravimetric analysis, see Figure 2. At 800°C the tungsten in Pt-8.5W alloy underwent vigorous
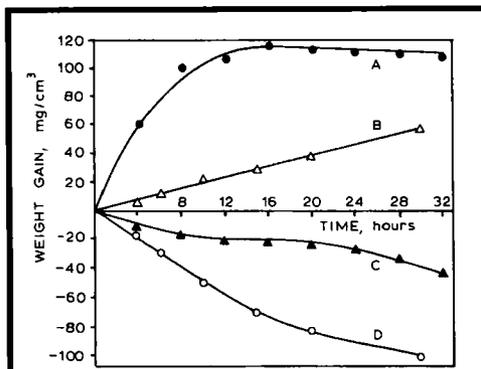

Fig. 2 The weight gain or loss for a series of platinum-tungsten alloy wires
A Pt-W-Re-Ni-Cr at 900°C
B Pt-W-Re-Ni-Cr-Y at 800°C
C Pt-W-Re-Ni-Cr-Y at 900°C
D Pt-8.5W at 800°C

evaporation, and the zero drift of the alloy became unacceptable (5). This alloy therefore cannot be used to measure strain at temperatures over 700°C.

For the Pt-W-Re-Ni-Cr alloy, during the initial 8 hours of oxidation, the weight gain curve is parabolic, with substantial weight gain, but on prolonged oxidation the curve assumes and maintains a horizontal line and with no further apparent gain or loss of weight, showing that the oxidation and evaporation of the alloy components have reached equilibrium.

On oxidation below 800°C, the Pt-W-Re-Ni-Cr-Y alloy, for use at 800°C, gains weight. However, when the newly developed Pt-W-Re-Ni-Cr-Y alloy, for use at 900°C, is oxidised at 900°C there is only a little weight loss during the initial stage; from 6 to 28 hours the curve remains nearly horizontal, but after 28 hours the weight loss tends to accelerate.

Structural Observations
[i] Metallographic Observation

The macrostructures of the alloy samples were observed under oxidation treatments. For both Pt-W-Re-Ni-Cr and Pt-W-Re-Ni-Cr-Y a film appeared after a 20 minute oxidation treatment.

The Pt-W-Re-Ni-Cr alloy still retained the inner recovered structure. On prolonged oxidation, the thickness of the oxidised film increased and the surface became irregular. After a 10 hour oxidation, surface cavities appeared, due to some of the oxide particles peeling off. Oxidation follows the grain boundaries and forms many corrosive spots on the grain boundaries, and the grains grow in size. The thickness of the oxidised film is ~ 20 μm.

In contrast, after the Pt-W-Re-Ni-Cr-Y alloy underwent oxidation for 5 to 10 hours the surface layer became a continuous film, 7 to 10 μm thick. Internal oxidation was hardly noticeable, and the grain size was almost unchanged.

[ii] SEM and EDS Analysis

SEM images of the surface topography and the corresponding area and line scans for the Pt-W-Re-Ni-Cr sample are shown in Figure 3. After oxidation for 30 hours, the surface texture

was quite different to the inner texture. The surface had been oxidised to a very thick layer. Area analyses of element distributions showed that nickel had the highest surface concentration of all the components, and had a continuous distribution to a depth of about 10 μm, Figure 3(a). Chromium was also concentrated on the surface layer but at a lower concentration than nickel, Figure 3(b). Tungsten was more uniformly distributed in the grains compared to nickel and chromium, and had a little higher concentration on the surface, Figure 3(c).

Similar results were obtained during line analysis of the elements. Tungsten, nickel and chromium components concentrate on the surface layer; a "component-free" layer (without tungsten, nickel and chromium) then appears between the surface layer and the inner part. This shows that the oxidation is a diffusion controlled process. Rhenium showed a very constant distribution throughout the wire, except for a slightly lower surface concentration.
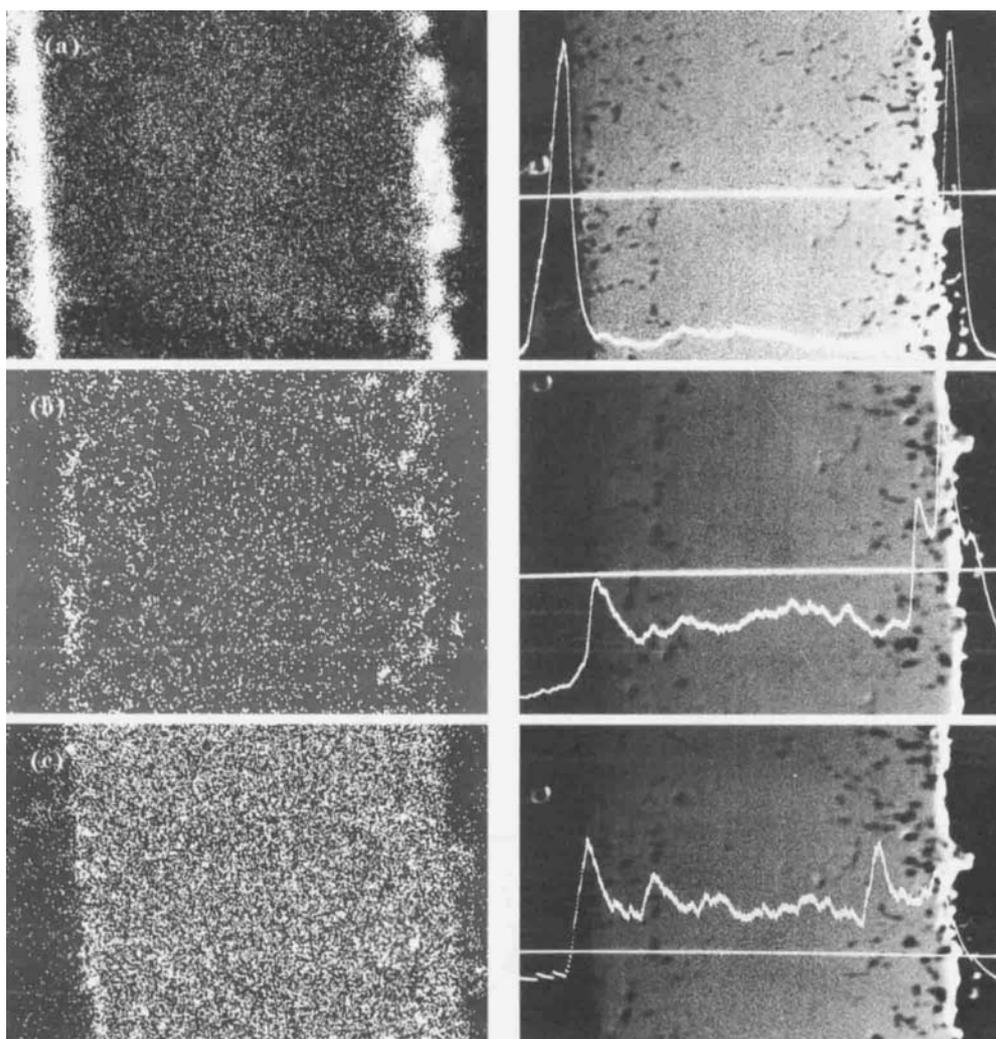

Fig. 3 The micromorphology of the Pt-W-Re-Ni-Cr alloy after oxidation for 30 hours at 900°C, showing the distribution of the elements; area scans are on the left and line scans on the right: (a) nickel (Kα); (b) chromium (Kα); (c) tungsten (Mα)   magnification is approximately ×510
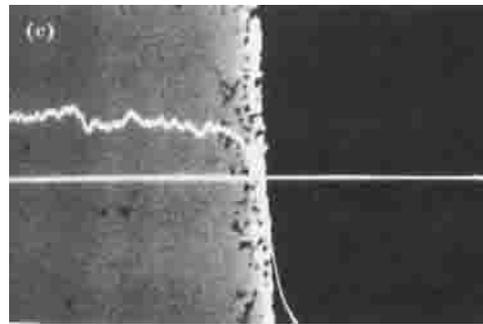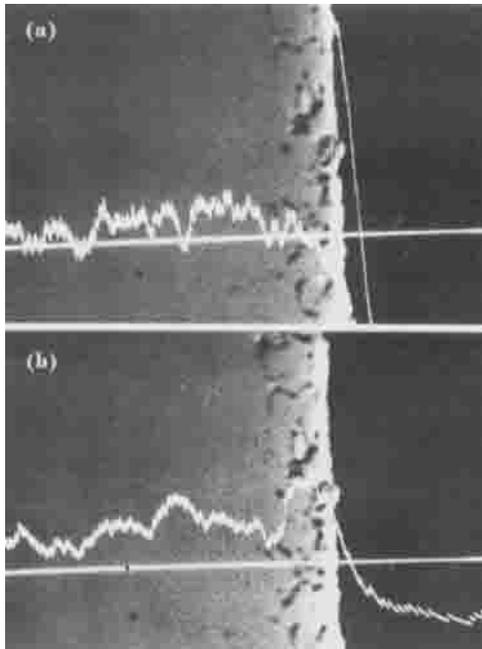
Fig. 4 Line scans of the newly developed Pt-W-Re-Ni-Cr-Y alloy after oxidation for 30 hours at 900°C and the corresponding element distribution:
(a) nickel (K_α)
(b) chromium (K_α)
(c) tungsten (M_α) magnification × 510

The surface features of the Pt-W-Re-Ni-Cr-Y sample are similar to those of Pt-W-Re-Ni-Cr, see Figure 4. The surface oxidised film is more compact and thinner (7 ~ 10 μm), and the nickel and chromium concentrations are lower on the surface layer. The nickel and chromium "component-free" layer is not as conspicuous as in Pt-W-Re-Ni-Cr alloy. Tungsten and rhenium are evenly distributed, except tungsten has a slightly lower concentration on the surface layer.

[iii] Transmission Electron Microscopy

The Pt-W-Re-Ni-Cr-Y alloy was studied using TEM, see Figures 5 and 6. The grain boundary is distinct and no second phase particles are

observed. There are some dislocation networks in the as-drawn sample. Indexing of the diffraction pattern has confirmed that the two new alloy systems remain as platinum based solid solutions.

[iv] X-ray Analysis

Oxides appeared after a 20 minute oxidation treatment; this was also observed during the metallography. For the Pt-W-Re-Ni-Cr alloy, from the height of the diffraction peak and the order in which the phases emerged, the order of surface oxides was $NiWO_4$-$NiCr_2O_4$-NiO-CrO_2 (minimal) and from the quantity of the oxides, the order was $NiO > NiWO_4 > NiCr_2O_4$. After oxidation for 30 hours, the surface oxides were still NiO, $NiWO_4$ and $NiCr_2O_4$.

By adjusting the composition of the Pt-W-Re-Ni-Cr-Y alloy the oxide configuration was changed. If the tungsten and chromium
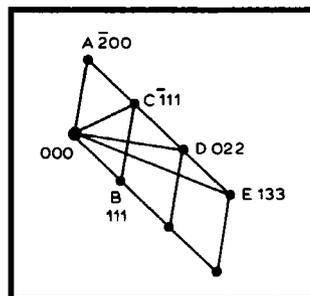

Fig. 5 Diffraction analysis shows that the newly developed Pt-W-Re-Ni-Cr-Y alloy is still a platinum based solid solution
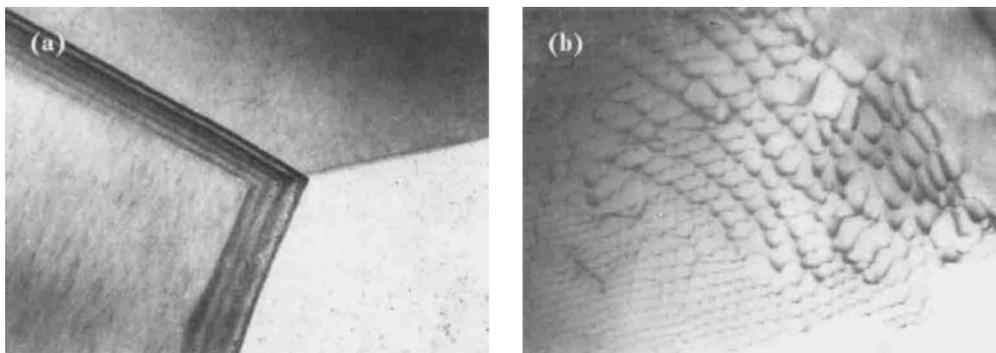
Fig. 6 TEM of the Pt-W-Re-Ni-Cr-Y alloy; (a) annealed; (b) as drawn

contents are increased, the nickel content decreased and yttrium is added, then $NiCr_2O_4$ becomes the dominant protecting layer. The others are $NiWO_4$, WO_3 and CrO_2 (the last two are present only in small amounts). With prolonged oxidation, the amounts of WO_3 and $NiWO_4$ gradually decrease, and after oxidation for 30 hours WO_3 has totally evaporated, and only a small amount of $NiWO_4$ remains. The final oxide configuration is: $NiCr_2O_4$-CrO_2 (small amount)-$NiWO_4$ (traces).

Strain Gauge Properties and Uses

The structures, thermal output and the zero drift curves of the newly developed single wire strain gauge and the half-bridge strain gauge made of Pt-W-Re-Ni-Cr-Y and Pt-Ir-Ni-Cr-Y wires are shown in Figures 7 to 10.

The zero drifts of the Pt-W-Re-Ni-Cr-Y and the Pt-Ir-Ni-Cr-Y alloys are smaller than that of Pt-W alloy, and are of positive value. Between room temperature and 900°C, the temperature compensation is perfect and the average thermal output of the half-bridge strain gauge, made of the Pt-W-Re-Ni-Cr-Y and Pt-Ir-Ni-Cr-Y, keeps very good convergence in the low temperature range. The reproducibility of three subsequent thermal cycles to 900°C are within 200 microstrains.

This half-bridge strain gauge, which was developed by the Beijing Institute of Aeronautics and Astronautics, has been put to practical use in aircraft failure analysis in China. The first stage working turbine blade of a civilian airplane fractured repeatedly causing cancellation of civil flights. The dynamic stress within the turbine blades was measured using the strain gauge, under the working temperature conditions of 600 to 700°C, and at over ten thousand rotations per minute. The recorded data were comprehensive, accurate and reliable, and provided the major basis for failure analysis and solution of the problem.

Discussion

After holding samples at 1400°C for 256 hours, the Pt-(8~40)W alloy still remains as a solid solution (9). When rhenium, chromium and nickel were added to the Pt-W alloy, the new Pt-W-Re-Ni-Cr and Pt-W-Re-Ni-Cr-Y alloys remained as solid solutions.

When Pt-8.5W alloy is held at 800°C for 100 hours an irregular surface is visible, and no protection layer has formed. The oxidation has travelled into the interior along grain boundaries, forming many corrosive spots on them.

On addition of oxidation-resistant nickel and chromium the surface topography of Pt-W-Re-Ni-Cr and Pt-W-Re-Ni-Cr-Y alloys is changed, by growth of the comparatively compact oxidised surface layer. The alloys generally retain their fine grain structure and the inner texture is almost unaffected by the oxidation. The nickel and chromium preferentially oxidise forming a compact film on the surface, which protects the base alloy from further oxidation.

The ion radius of nickel is 0.67 Å – similar to that of platinum, tungsten and rhenium. Nickel has the lowest melting point of all the added components, which gives it a comparatively high diffusion rate and thus preference in forming
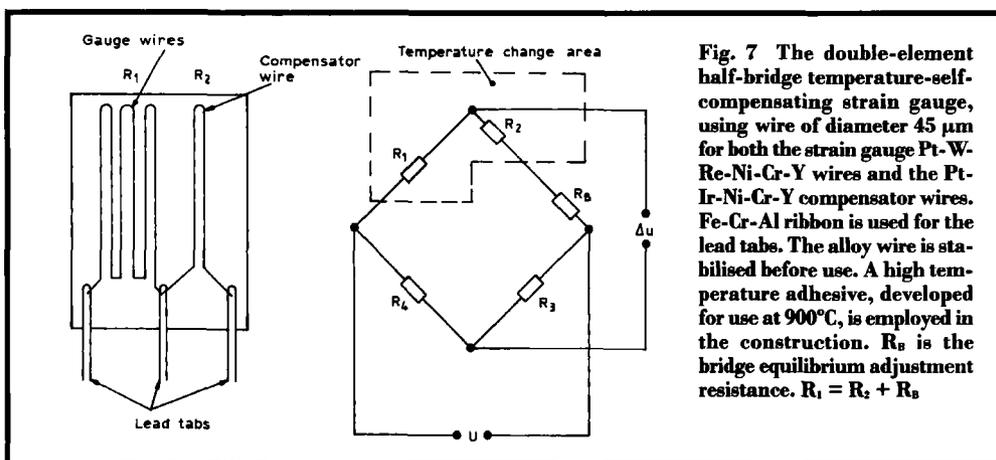
Fig. 7 The double-element half-bridge temperature-self-compensating strain gauge, using wire of diameter 45 μm for both the strain gauge Pt-W-Re-Ni-Cr-Y wires and the Pt-Ir-Ni-Cr-Y compensator wires. Fe-Cr-Al ribbon is used for the lead tabs. The alloy wire is stabilised before use. A high temperature adhesive, developed for use at 900°C, is employed in the construction. R_B is the bridge equilibrium adjustment resistance. $R_1 = R_2 + R_B$

a thick, non-porous and compact NiO film. As the volume ratio NiO:base alloy becomes greater than 1, ($V_{NiO}:V_{Pt} = 1.4$), the NiO surface film will completely cover the base alloy and be compact, Figure 3(a).

Generally, the presence of both NiO and Cr_2O_3 results in the formation of the more stable spinel-type $NiCr_2O_4$, that is $NiO + Cr_2O_3 = NiCr_2O_4$ (10), and with the preferred formation of $NiWO_4$ for the Pt-W-Re-Ni-Cr alloy, the surface oxide configuration is as follows:

(a) first protection layer is the $NiWO_4$ salt with melting point above 2000°C

(b) second protection layer is a thick and compact p-type semiconducting NiO film

(c) third protection layer is the most stable $NiCr_2O_4$.

The formation of these oxides results in a conspicuous weight gain during the initial stages of oxidation, see Figure 2. After further oxidation of 6 to 10 hours, more oxide forms which makes it increasingly difficult for the Ni^{2+} and Cr^{3+} to diffuse to the surface for further oxidation. Therefore the oxidation process saturates and the oxidation rate noticeably decreases.

Over the time interval 8 to 30 hours the weight gain maintains a horizontal line. At this stage, evaporation of the alloy components has been checked. Evaporation of the surface oxides would cause a slow weight loss for the alloy and the semiconducting NiO would adjust the electrical resistance. However, there is a very small change in the alloy resistance and good linearity for the resistance/temperature relationship.

Adding yttrium to Pt-W-Re-Ni-Cr thins the grain size, which remains stable, even after oxidation for the same time as the Pt-W-Re-Ni-Cr alloy. Figure 2 shows that during the initial 4 hour oxidation the alloy sample losses weight, because the protecting oxide film has not yet formed and the tungsten (of higher content in the Pt-W-Re-Ni-Cr-Y alloy) has evaporated.

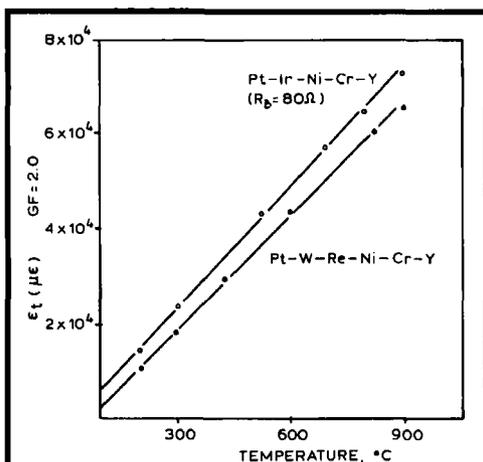For the Pt-W-Re-Ni-Cr-Y alloy, after the

Fig. 8 The average thermal output (or apparent strain change versus temperature) up to 900°C for the single wire strain gauge. G.F. is the gauge factor = $\Delta R/R/\Delta \varepsilon$; R_B is the bridge equilibrium adjustment resistance

oxidation treatment shown in Figure 2, the adhesion between oxide-oxide and oxide-base alloy is good, and oxide particles have not peeled off the base alloy. There are few cavities – which have not joined up. This has made diffusion of nickel, chromium and oxygen ions through the cavities difficult. Oxidation channels have been difficult to form, so the alloys possess very good oxidation resistance.

For the Pt-W-Re alloy held at 800°C, the resistance increased rapidly (5), due to the vigorous evaporation of tungsten and rhenium, and to the decrease in the alloy diameter. The rhenium becomes evenly distributed after nickel and chromium are added to the alloy and it is held at 900°C for 30 hours. The concentration and oxidation of the nickel and chromium on the alloy surface prevent rhenium evaporation. Thus, these two alloy systems maintain comparatively high strength and low TCR, which enables them to be used at 900°C.

The tungsten content in Pt-W-Re-Ni-Cr-Y and Pt-W-Re-Ni-Cr is higher than the rhenium content, and it has higher chemical activity; thus it reacts with nickel and chromium to form $NiWO_4$, Cr_2WO_4 and WO_3. These oxides will evaporate on prolonged oxidation, but at a much slower rate than the elements. This also ensures a long service life for the strain gauges.

The weight gain curves of Pt-W-Re-Ni-Cr-Y for use at 800 and 900°C, show that at 800°C it gains weight, but at 900°C it losses weight. Thus, wire used for strain measurements should be based on the 900°C alloy with an increased nickel content, the chromium content being adjusted according to the nickel:chromium ratio. The aim is to form a compact thick NiO film on the surface, and a stable $NiCr_2O_4$ film, which will prevent total evaporation of $NiWO_4$ from the surface. This alloy will be more stable with hardly any weight gain or loss during a long oxidation process, and it will fulfil the requirements for static strain measurement at 1000°C.

Conclusions

The newly developed Pt-W-Re-Ni-Cr and the Pt-W-Re-Ni-Cr-Y alloys possess a stable resistance/temperature relationship, a linear TCR/T
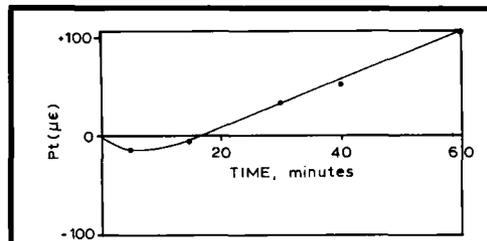

Fig. 9 The average zero drift (or apparent strain versus time) of a half-bridge gauge at 900°C
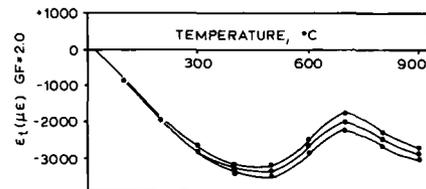

Fig. 10 The average thermal output of a half-bridge gauge

relationship and have a small temperature coefficient of resistance. The co-ordination between the Pt-W-Re-Ni-Cr-Y working wire and the Pt-Ir-Ni-Cr-Y compensating wire is good and the alloys have measured static strain satisfactorily over the temperature range 0 to 900°C.

At high temperatures the evaporation of tungsten and rhenium causes a drop in resistance, while a decrease in the alloy diameter increases the resistance. For the Pt-W-Re-Ni-Cr alloy, the formation of an ~ 20 μm thick oxide film has greatly decreased the effective area for electrical conduction; this causes the positive zero drift of the alloy. However, for the Pt-W-Re-Ni-Cr-Y alloy, the thinner oxide film (7 to 10 μm) and evaporation of some oxides causes a negative zero drift for the alloy. Adjusting the composition will produce an alloy in which the fall in resistance, due to evaporation, is exactly matched by increased resistance, due to a decrease in the effective area of conduction, from oxide film formation. The zero drift of the alloy will be very small and the maximum usable temperature can be extended to at least 1000°C. Such gauges will enable strains in aircraft engines and rockets to be checked more consistently and accurately, ensuring greater air travel safety.

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Sulfur-Resistant Platinum Hydrogenation Catalysts

There is increasing awareness of the damaging effects on human health caused by the pollution from diesel fuelled vehicles, especially the harm due to the presence of particulate matter of 10 μm or less. The aromatic particulates content of diesel fuel is known to be a primary source of pollution, and legislation and research have been directed towards reducing the levels in diesel fuels (1).

In order to comply with new specifications, petroleum refiners may use an additional hydrotreating procedure, usually with a relatively inactive metal sulfide catalyst, which requires severe operating conditions. Alternatively a two-stage process can be used, the second-stage being hydrogenated by very active noble metals. However, catalyst poisoning by the sulfur compounds in the petroleum feedstock is a problem, and a first stage hydrotreatment is needed to reduce the sulphur to below 10 ppm.

Researchers from CANMET in Canada have now studied two molecular sieve catalysts, impregnated with platinum, for their resistance to sulfur poisoning during hydrogenation of the aromatics of synthetic crude distillates (2). The molecular sieves they examined were a sodium montmorillonite, made into a pillared inter-layered clay (PILC), and Y-zeolite. The feedstocks used were middle distillate fractions from a synthetic crude oil, from Canadian oil sands, which have significantly different properties to conventional fuels.

The Y-zeolite had a much higher microporosity than the PILC, while the parallel-plate formations in the PILC create a unique structure with interior galleries having well-dispersed ion-exchange sites.

They found an acceptable hydrogenation activity for platinum clusters supported in Y-zeolite, when a distillate feedstock, containing 140 ppm sulphur and 50 ppm nitrogen, was upgraded. There was also an improved poison resistance to the feedstock, which contains sulfur levels at least an order of magnitude higher than required for conventional catalysts. The platinum/Y-zeolite catalyst functioned at temperatures signifi-cantly lower than those for conventional hydro-treating catalysts with superior activity for the hydrogenation of middle distillate aromatics. This is attributed to the structure of the Y-zeolite supercage, producing well-dispersed electron-deficient platinum metal clusters. Although the platinum/PILC catalyst had no enhanced resistance to sulfur poisoning, its unique structure may be useful in ring opening reactions for naphthene in middle distillate feedstocks.

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Sulfur Removal by Platinum

Petroleum and natural gas contain sulfur, and its removal by hydrodesulfurisation produces hydrocarbons and hydrogen sulphide, which may be removed via the Claus process. In the Claus process, sulfur is recovered from hydrogen sulfide in a two-stage process in which hydrogen sulfide is mixed with oxygen to yield water and sulfur dioxide, the latter then reacts with more hydrogen sulfide to produce sulfur and water.

Researchers from McGill University, Canada, have now produced a homogeneous platinum catalyst which mimics the Claus process (A. Shaver, M. El-khateeb and A.-M. Lebuis, *Angew. Chem., Int. Ed. Engl.*, 1996, **35**, (20), 2362–2363). They report on the reaction of *cis*-[(PPh₃)₂Pt(SH)₂] (*cis*-2) with sulfur dioxide, to produce [(PPh₃)₂PtS₃O] (1). Both complexes catalyse the Claus reaction.

The catalytic cycle proposed for this reaction involves adduct formation between *cis*-2 and sulfur dioxide, followed by sulfur dioxide insertion into the sulfur-hydrogen bond. Elimination of water produces (1), which reacts with hydrogen sulfide to cleave the platinum-sulphur bonds and regenerate *cis*-2. The other cleavage product was not detected, and may be a key intermediate in the process.