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DEVELOPMENT OF ADVANCED CERAMIC SPRINGS

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ABSTRACT

This paper describes the results of a collaborative research project in which a range of ceramic springs were developed which could be reliably used in conditions under which metallic springs would fail: for example in high temperature applications, and/or corrosive and oxidising conditions, Using several different process methods like green machining, extrusion and injection moulding, both coil and disc springs were manufactured from four different ceramic materials; silicon nitride, magnesia stabilised zirconia, yttria stabilised zirconia and alumina hardened zirconia. The maximum service stress and operating temperature was defined for ceramic springs, made from each of the afore named materials.

INTRODUCTION

The primary objective of the project was to expand the range of engineering conditions in which mechanical springs may be reliably utilised. Metallic springs start to collapse at temperatures above 550°C, whereas ceramic springs have potential for use at temperatures up to 1000°C or higher. A second limitation in the current range of conditions in which metallic springs may be used is corrosion and oxidation resistance, even when the highest performance nickel alloys are used. Some advanced ceramic materials offered clear potential for springs that are used in certain corrosive or highly oxidising conditions, particularly at high temperatures. The object of this project was to expand the operational range of springs into high temperature (500° - 1000°C) and **highly** corrosive environments by exploiting the material properties of ceramics, in particular yttria stabilised zirconia, zirconia toughened alumina and silicon nitride. Ceramic springs have been developed in Japan [1] and production processes patented [2][3]. Ceramic springs are available from Japan in both silicon nitride and in partially stabilised zirconia and as a consequence of this research, similar coil and disc springs are now commercially available in Europe.

A secondary objective of the project was to develop spring manufacturing process routes for advanced ceramic materials such that these processes would be beneficial for the production of components other than springs. Springs are very highly stressed components - usually the highest stressed in any manufactured engineering product - and so demand a capability of withstanding high stresses at their surface without significant risk of sudden catastrophic failure. Hence manufacturing process routes developed for ceramic spring products would enable the full potential mechanical properties to be established that could be exploited in other components.

MATERIAL EVALUATION

The theoretical flexural strength of advanced ceramics is very high but their structural uses have been limited in the past by a brittle fracture mode which is readily initiated from quite small defects. Often large defects result from the most commonly used forming processes, such as powder pressing, slip casting, extrusion and injection moulding. One major source of defects are the agglomerates inevitably present in fine powders as a result of van der Waals forces. Since springs require the full strength properties of the ceramics to be realised, especially at the surface, process routes for spring manufacture had to be devised whereby high integrity macro defect free structures were produced.

Four advanced ceramic materials were identified as having potentially attractive spring properties namely; sintered silicon nitride (Si_3N_4), magnesia stabilised zirconia (MgO PSZ), yttria stabilised zirconia (Y_2O_3 PSZ) and alumina hardened zirconia (AHZ). The advantages and disadvantages of each material in terms of spring manufacture are discussed in further detail below.

Silicon Nitride (Si_3N_4)

Sintered silicon nitride with Y_2O_3 and Al_2O_3 additions is a high strength (1000 MPa) and low density (3.3 g/cm^3) ceramic which makes it very interesting for spring manufacture. However, a potential disadvantage for springs, is the lower integrity microstructure of the as-sintered surface layer which silicon nitride exhibits. The as sintered surface has a much lower strength than the sintered core material, and therefore machining a minimum of 100 microns of the as sintered surface becomes a necessary operation to allow the maximum safe design stress to be utilised. The role and nature of the as-sintered surface layer on sintered silicon nitride was established and the process route associated with the maximum strength of the as-sintered surface was identified,

The critical surfaces of disc springs could be machined, but for coil springs, the machining of the critical spring surfaces was difficult and expensive to accomplish, so therefore the maximum design stresses for silicon nitride coil springs have to reflect the strength of the as sintered skin. Two different sintering methods were employed during the project; sintered the springs under a gas pressure of 10 MPa and a presureless sintering method, both giving similar strengths for the as-sintered skin. It was found that some improvement in the integrity of as sintered surfaces can be achieved by a barreling process in which springs are tumbled with ceramic abrasive media.

Silicon nitride retains its strength up to and above 1000°C, but when springs were used in oxidising conditions at high temperature (>900°C), some loss of strength was observed. Two independent studies by partners in this project showed that the strength of the material is degraded slightly above 900°C. Hence silicon nitride could not be used at its maximum design stress for long term applications above 900°C in oxidising conditions.

Magnesia Stabilised Zirconia (MgO PSZ)

“This material has relatively modest bend strength values at room temperature (600 MPa maximum), but this value was consistently and reliably achieved when manufacturing springs. Zycron L was selected as the standard material for magnesia stabilised zirconia, due to its good green strength and machinability. It has relatively low E and G moduli, compared with other ceramic materials (low modulus values enable maximum energy storage). Testing at elevated temperatures (>600°C) revealed that the reduction in usable strength was quite small.

Zycron L coil springs were generally manufactured by green machining. However, an injection moulding mix was also developed using a thermoplastic binder. The development of this mix allowed for a direct comparison of springs manufactured from the green machining and the injection moulding route.

Yttria Stabilised Zirconia (Y_2O_3 PSZ)

This material has a relatively high bend strength value at room temperature (≈ 1100 MPa), and these values were regularly achieved using a number of different spring manufacturing routes. The tetragonal to monoclinic transformation which affords this material its strength at room temperature, starts to be lost above 400°C. Nonetheless the strength retention of yttria stabilised zirconia at temperatures between 600° and 850°C appears to be similar to that of magnesia stabilised zirconia. Both the strength and density of yttria hardened zirconia can be improved by Hot Isostatic Pressing (HIPping) after sintering.

Yttria stabilised zirconia was processed into springs either by viscous polymer processing (a process patented by ICI), or by the green machining of a 'press mix', the green strength of which was quite high. The material was also available for injection moulding using a thermoplastic binder mix. The development of this mix allowed for a direct comparison of springs manufactured by three completely different process routes.

Alumina Hardened Zirconia (AHZ)

At the outset of this reasearch project it was envisaged that zirconia toughned alumina (ZTA) would be used, but zirconia with a 20% alumina addition *(the so* called alumina hardened zirconia) was identified as having potentailly higher strength and lower modulus values than ZTA, and so AHZ was used in preference to ZTA. Initial trials resulted in springs which had too low a density and consequently poor strength results. Further trials to correct this problem using HIPPING at 1400°C, after sintering enabled estimated bend strengths of 1500 MPa to be achieved, but not the claimed maximum strength of> 1800 MPa.

SPRING MANUFACTURE

'The main requirements during the project were to develop process routes which would reduce and/or eliminate the incidence of surface and internal defects in the material, and also the amount of distortion that arose from sintering. A secondary requirement was that the process route was economical, if possible, more so than green machining.

Coil Spring Forming

During the course of the project, coil springs were manufactured using a number of different process routes;

- Green machining a thread into a tube and final reaching to a spring after sintering (AHZ was also hipped)
- Isostatic pressing to final shape and sintering
- Injection moulding a ceramic 'wire' to a spring and sintering
* Coiling a thermoplastic ceramic 'wire' to a spring and sintering
- Thermoplastic extrusion and sintering
- VPP, extrusion and sintering

Green Machining

This process has been successfully used for the magnesia and yttria stabilised zirconia and also the alumina hardened zirconia coil springs. Initially green machining was accomplished rather slowly and approximately on a lathe, but later in the project greater accuracy and speed was achieved by using a numerically controlled miller for green machining. Green machining allows tight tolerances to be achieved, but a disadvantage of the process is the loss of expensive raw material during green machining and the machining that is required after sintering which can be very time consuming. Trials were conducted to maximise the green machining and to minimise the post sinter machining.

A number of post-sinter machining methods were evaluated, notably drilling, laser machining and grinding for coil springs. Consequently the time required for post sinter machining was reduced, without significantly damaging spring performance, but all trials to eliminate the post-sinter machining were a failure. Nonetheless, development was sufficient that the green machining process route can now be considered as a potential y viable route for small quantities of coil springs.

Injection Moulding

Injection moulding using a thermoplastic mix was found to be a feasible method for the manufacture of yttria stabilised zirconia, magnesia stabilised zirconia and silicon nitride coil springs. Injection moulding is an economically process method for producing parts in larger quantities with more complicated shapes. Large quantities per design are essential to offset the high tooling costs. Limitations exist in regard to the developed spring length to wire diameter ratio, thereby reducing the number of allowable coils, a ratio of 1:100 is the maximum achievable. It is thought that this would be improved during continuous production of ceramic springs, with some further development of the flow properties of the injection moulded mix. It is expected that this manufacturing method maybe suitable for mass production of springs, although the spring performance has not yet been fully evaluated.

Thermoplastic Coiling

This is a possible process route for ceramic spring manufacture, but problems have not been fully solved of lamination defects which cause the spring to fail at rather low stresses. The number of coils was limited by injection moulding and so the thermoplastic coiling process was developed, which has the significant advantage that no mould is required. A ceramic/binder mix was developed that had sufficient thermoplastic properties to coil a rod into a spring shape. Using this method, no limitation exists for the diameter to wire ratio. The process is very similar to the production of metallic springs, which are heat treated after forming.

Initial trials were conducted, although *a* problem was identifed concerning the precise control of temperature during thermoplastic coiling. The temperature has to be high enough for the ceramic rod to exhibit good plastic behaviour, but still low enough to avoid the ceramic sticking to the support and/or stretching the ceramic which could result in breakage. Another problem which was encountered during the coiling trials was that the inner side is compressed whilst the outer side is stretched, the latter it was thought may cause structural defects in the springs resulting in early failure. Springs manufactured by this process still need further evaluation.

Viscous Polymer Processing

This process method was demonstrated to be a feasible and economical manufacturing route for smaller dimension coil springs with closed ends, more so if the distortion problems could be overcome. Distortion is a common problem to all manufacturing processes except green machining, and as such will be discussed in further detail below. The problem of longitudinal cracking was almost eliminated by optimisation of the dough formulation and extrusion parameters.

Manufacturing springs in extruded sections larger than 2mm led to problems but this was overcome by increasing the extrusion die to wire ratio and by being patient and not accelerating the drying process. The forming of closed and ground ends was achieved in the green state, and grinding of the ends was accomplished after drying but before sintering.

Table 1
Manufacturing Methods

	springs Disc	springs Coil	Advantages	Disadvantages
Isopress, green machining & post-sinter machining	✓	✓	Very accurate Low tooling costs Large spring sections are possible	Slow process High material 10ss
Isopress	-	-	Not feasible	
VPP & extrusion	-	✓	Cheaper than machining Closed ends possible	Distortion Larger ssections are crack prone
Injection moulding	-	✓	Suitable for large	Too short a length for most springs High tooling costs
Thermoplastic winding	-	✓	Closed ends possible	Susceptible to cracking Distortion
Thermoplastic extrusion		✓	Suitable for large quantities	Process development required to overcome delamination

Disc Spring Forming

During the course of this project, green machining was developed to be an efficient process for the manufacture of ceramic disc springs. Manufacturing disc springs from a sintered solid body was , found to be both time consuming and expensive, but was a useful method for 'first' springs, which were used to estimate the maximum deflection and hence spring properties during the first year of the project.

The most cost effective forming method for ceramic disc springs 'was found to be green body technology. However, it was found that if the disc springs are to be sintered to the final dimensions, then the surfaces that were to be stressed in tension had to be free of flaws, as these would cause failure. Hence silicon nitride disc springs always had to be post-sinter machined to remove about 100 microns on the inside, outside and lower side.

The larger disc springs manufactured from magnesia and yttria stabilised zirconia did not require post sinter machining except to skim the top and bottom bearing surfaces so as to avoid point loading.

Sintering

The final properties of a ceramic are achieved by sintering. The heat up conditions, the maximum sinter temperature, the dwell time and the cooling curve all influence the crystallisation process and grain growth, and in the case of yttria stabilised zirconia are responsible for the transformation toughening effect.

During the first year high levels of distortion were experienced by each spring partner, although it was still possible to evaluate the spring performance, even though the springs were not commercially or technically acceptable.

The use of a central mandrel allowed uniform linear shrinkage of the outside diameter but it was found that the coil springs still tended to unwind during sintering resulting in the outside diameter being larger at either end than in the middle. Another problem was the uneven coil to coil pitch. However, this could be reduced by the use of a grooved mandrel, of which a number of designs were tried, the most innovative being a reusable multi-piece graphite former. By sintering the springs on a former made out of the same material as itself, it was possible to get almost undistorted springs but this process was found to be uneconomical because the former had to be destroyed in order to remove the spring from it. The distortion problem with disc springs was overcome by using *a* fluidised bed rather than mechanical means of support for *green* machining parts during sintering.

Even though the sintering process was vastly improved during the project, there was still the probability that the prospective end user would have to accept wide load tolerance limits at the present stage of development, with all except the green machined springs.

In order to achieve maximum strength in the alumina hardened zirconia materials, hot isostatic pressing (kIIP), was required. An increase in density was achieved after HIPping, during which the zirconia changed colour from white to grey or even almost black. This colour change was due to the reducing condition during the HIP cycle. From the results obtained, there appeared to be benefit gained from hipping the alumina hardened zirconia springs, and this material and process route offered the best chance of success for the highest stress or dynamic spring applications. Hot isostatic pressing of magnesia stabilised zirconia had a detrimental effect upon the strength and density, with significant reduction observed, especially in the latter.

The silicon nitride disc springs could be sintered satisfactorily using a standard sinter cycle, developed during the first year. It was found to be important, however, to sinter the springs in the same part of the furnace, for an even temperature distribution, so that reproducible springs could be manufactured.

Fig 1 shows the extensive range of ceramic springs that have been successfully made during the course of this project. The photograph clearl y shows the wide range of spring sizes that can be manufactured. The ‘round’ wire compression springs have been manufactured by either extrusion or injection moulding process routes, whereas the ‘rectangular’ section springs at the back of the photograph have been made by the green machining process route.

TEST PROCEDURE

Springs were tested in the laboratory using a combination of the following techniques, so as to evaluate the spring properties.

- Load Testing
  The springs were load tested accurately to determine the spring rate, and sometimes the load at which fracture would occur. The data so obtained was used to estimate the equivalence of load and stress by use of CAD prograrnmes, especially adapted for ceramic materials,

- Fatigue Test
  Coil springs made from various ceramic materials and disc springs from silicon nitride were fatigue tested. The springs were tested at speeds of approximately 1/13th of their natural frequency, at which no apparent resonance occurred.

- High Temperature Load Testing
  Springs were load tested at 650°C and in some cases also at 900°C. The aim of this test was to determine the change in the spring rate at high temperature and to check whether a reduction (was applicable) in the maximum stress at which springs would operate.

- Relaxation Testing
  Springs were selected which had similar free lengths and spring rates at ambient temperature. The springs were placed under constant load to achieve a stress of approximately 100 MPa at 800°C for 24 and 72 hours, the spring rate and free length were checked after each test to check for susceptibility to loss of Ioad at temperature i.e. relaxation. The test was then repeated using metallic Nimonic 90 springs for comparison.

- Sustained Load Test
  The purpose of this test was to see what effect, if any, a sustained load would have upon both coil and disc springs, i.e. susceptibility to slow crack growth. A selection of springs of all materials were load tested to a stress just below their maximum stress and then left for approximately 17 and 66 hours.

- Corrosion Testing
  The aim of these tests were to determine the corrosion resistance of the ceramic materials under salt spray conditions.

  Samples of the magnesia stabilised zirconia coil and disc springs, silicon nitride disc springs and yttria stabilised zirconia coil springs, were subject to a 72 and 168 hour salt spray test, with intermediate inspections after 24 and 48 hours. The springs were then re-load tested.

RESULTS

The disc springs were load tested to determine the maximum stress each material would withstand at **room** temperature without significant risk of fracture, the results of which are shown in Table 2..

Table 2

Spring Type	Material	Process	Max. Tensile Bending Stress / MPa
Disc	Si_3N_4	Fully Machined	630-650
Disc	Si_3N_4	Part Machined	540-640
Disc	Si_3N_4	Not Machined	220-300
Disc	MgO PSZ	Not Machined	300
Disc	Y_2O_3 PSZ	Not Machined	300

These results clearly show the effect of the as-sintered skin on the performance of silicon nitride disc springs. The part machined silicon nitride disc springs were machined only on the surfaces stressed in tension. As will be seen from table 3, the PSZ springs performed satisfactorily without post sinter machining,

Table 3

Spring Type	Material	Process	Max. Torsional Stress/ MPa
Coil	Si_3N_4	Extrusion	<200
Coil	Y_2O_3 PSZ	VPP/Extrusion	**390**
Coil	Y_2O_3 PSZ	Green Machined	**420**
Coil	Y_2O_3 PSZ	Injection Moulded	**200-400**
Coil	MgO PSZ	Green Machined	**230**
Coil	MgO PSZ	Extruded/Coiled	**200**
Coil	MgO PSZ	Injection Moulded	**230**
Coil	AHZ	Green Machined	**330**
Coil	AHZ	Green Machined/Hipped	**460**
Coil	AHZ	VPP/Extrusion	**300**

It could be seen from the above results that both injection moulding and extrusion were methods which could be capable of producing springs with similar properties to green machined springs, However, the internal defects, especially in the injection moulded springs were found to be a problem, although it is anticipated that improvements in the material flow process during injection could overcome this. Clearly the AHZ hipped springs gave the highest performance.

Fatigue Testing

The table below summarises the test results obtained.

Table 4

Spring	Material	Process	Fatigue Test Range/MPa	Comments
Coil	MgO PSZ	Green Machined	**25-150**	
Coil	Y_2O_3 PSZ	VPP/Extrusion	**50-200**	
Coil	Y*03 PSZ	Green Machined	100-300	no failures
	Y_2O_3 PSZ	Green Machined	190-370	all failed
Coil	AHZ	Green Machined	150-350	no failures
Coil	AHZ	Green Machined. Hipped	35-27mm	4/8 failed
Coil	AHZ	Hipped	190-370	
Disc	Si_3N_4	Green Machined	0.3mm stroke	no failures
	Si_3N_4	Green Machined	0.4mm stroke (100-600hIPa)	fatigue failures

High Temperature Testing

One advantage of ceramics is their ability to perform at elevated temperatures i.e. up to 900°C, at which temperatures metallic parts would collapse, the results obtained are shown below in table 5.

Table 5

SPRING	23°C				600°C		
	Max δ mm	Max Load N	Max Stress MPa	G Modulus MPa	Max δ mm	Max Load N	Max Stress MPa
Y_2O_3PSZ	11.60	84.7	290	73500	10.58	64	219 *
Y_2O_3PSZ	13.78	87.9	384	74500	6.63	38	166
Y_2O_3PSZ	8.96	78.2	277	80400	7.45	46	163 *
Y_2O_3PSZ	10.91	106.3	2i7	71000	9.45	73	149 *
Y_2O_3PSZ	13.35	154.3	319	90500	10.26	110	227 *
MgOPSZ	7.10	302.0	188	66000	5.50	151	94 *
AHZ	14.15	149.7	464	89100	11.40	108	335 *
AHZ	13.35	82.6	300	90000	6.84	37	134

* indicates failure at high temperature

Relaxation Testing

Results obtained showed that the overall spring rate of each spring had apparently increased slightly, but that the free length had decreased.

Test Conditions : Temperature 800°C, Stress 100 MPa

Table 6

Spring	Free Length/mm			% Loss of Load	
	Ohr	24hrs	72hrs	24hrs	72hrs
Nimonic 90	28.79	27.35	25.21	33	83
MgO PSZ	47.63	46.61	44.61	3	6
Y_2O_3 PSZ	42.43	40.97	40,77	17	22

Sustained Load Testing

The results obtained showed that only one failure occurred, in a magnesia stabilised coil spring. The failure occurred between 17 and 66 hours, from art internal defect, and so it would appear that this failure was an isolated case, but nonetheless it illustrates that slow crack growth will occur even at the relatively low stresses to which these springs were subject.

Corrosion Testing

The aim of these tests were to determine the corrosion resistance of the ceramic materials to salt spray testing.

The springs were reload tested after salt spray exposure arid it was found that the spring rate was unaffected, As expected no change in the spring appearance was noted as a consequence of these tests.

FAILURE INVESTIGATION

Throughout this project a number of springs have failed during manufacture or laboratory testing, For the most part it has been apparent as to why the springs have failed, as the pores, cracks or contaminants were visible with the naked eye. However, the process routes have improved such that , the number and size of defects have been significantly reduced, and so the need for further investigation has arisen. The fracture surfaces were examined by using either a low power optical microscope or the scanning electron microscope (SEM), depending upon the size and type of fracture. Spring samples, were sectioned using a diamond wheel to produce fracture surfaces small enough for examination on the SEM.

The majority of springs failed at more than one position so it was sometimes difficult to determine the fracture origin, In most cases, failure initiated from either an internal defect such as a pore or **from** external defect such as cracks, flaws or surface contaminant, examples of which are included in Figs 3-4.

The silicon nitride disc springs were often the most difficult of the ceramic springs to examine. Examination of the part machined disc springs revealed a skin microstructure as shown in Fig 5, from which an approximate thickness of each layer could be measured as shown below.

Skin	85pm
transition zone	350μm
core	740μm

It is believed that the lower strength of the skin was the main reason for failure.

Examination of the springs which failed on fatigue test, revealed a failure mechanism which was similar in appearance to a fatigue failure in metallic springs, as shown by Figs 6 and 7. It would appear that the crack has propagated and stopped several times before failure occurred.

CONCLUSIONS / SIGNIFICANT RESULTS

1. Material processing techniques for advanced ceramic materials have been developed for spring production.

2. Machining techniques, green and as-sintered, have been speeded up to make them economically viable.

3. The maximum stress design limits for static and dynamic conditions have been defined for a range of advanced ceramic materials, which were made into springs, test samples from which were shown to have mechanical strength values equal to the published values for each material. The engineering properties and capabilities of all the advanced ceramic materials considered have been much more accurately defined by the work in this project. When a material is claimed to have a static bend strength of 1500 MPa, it is now much clearer which range of strength can be reliably utilised in engineering components, of which springs are the most demanding.

4. Test methodologies for testing and handling of brittle spring materials have been established. A much better understanding has been acquired about the problems associated with manufacturing and use of open ended springs.

5. The influence of surface quality on susceptibility to failure has been defined.

6. The performance of the springs manufactured is close to that of similar products manufactured by Japanese companies. Ceramic springs have become commercially available from Japan during the course of this project. They are also available from Europe now, as a , consequence of this project, but the succesful widespread commercialisation of this product is thought to be five to ten years in the future.

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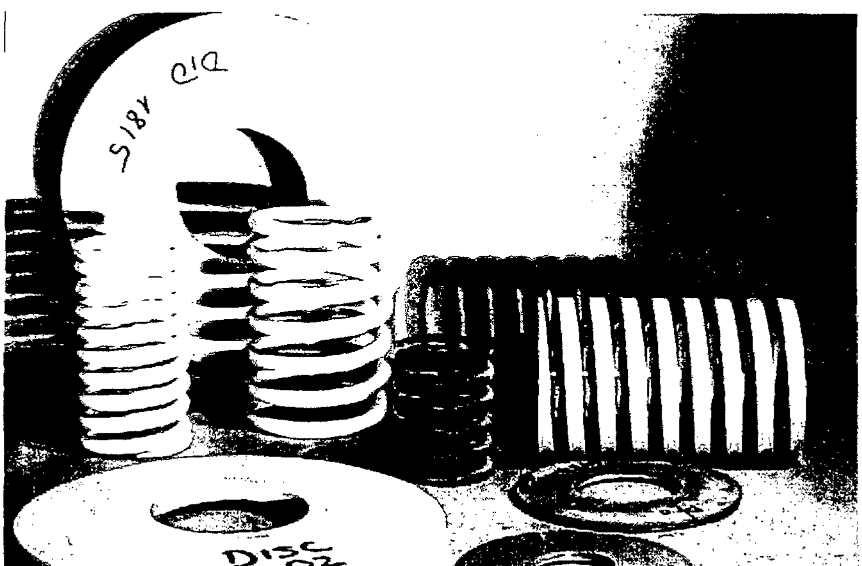
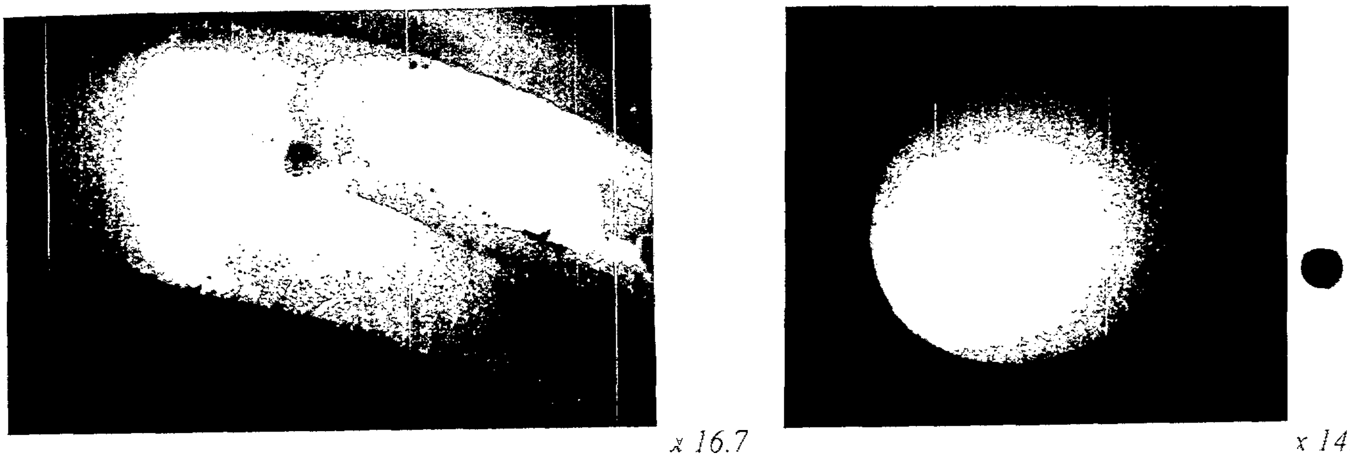
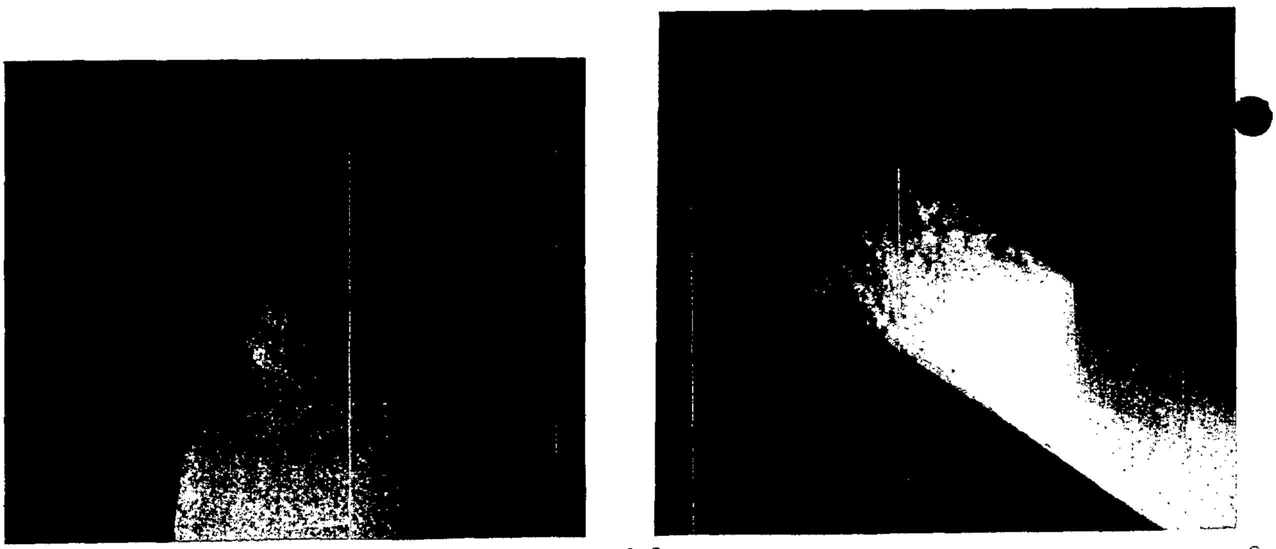


Fig 1 : Photograph showing the range of ceramic springs that have been manufactured during the course of this project.



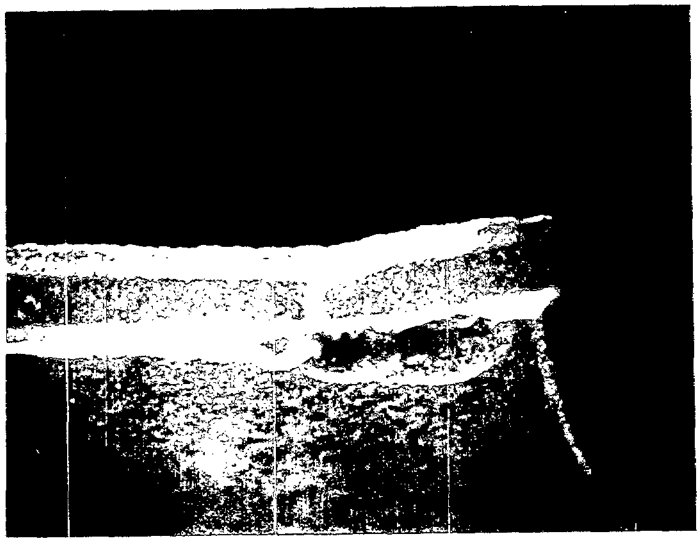
x 16.7 x 14.0

Fig 2 : Optical photographs showing detail of an internal defect which has initiated failure



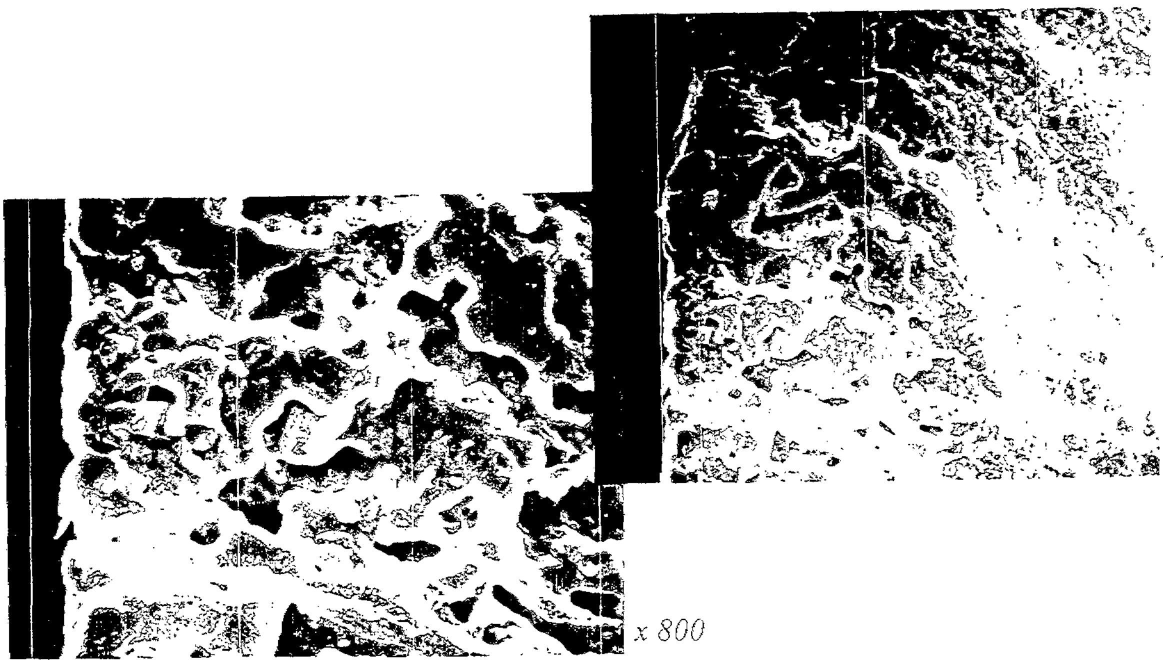
x 8.5 x 8.5

Fig 3 : Optical photographs showing detail of an external defect which has initiated failure.



x 11.3

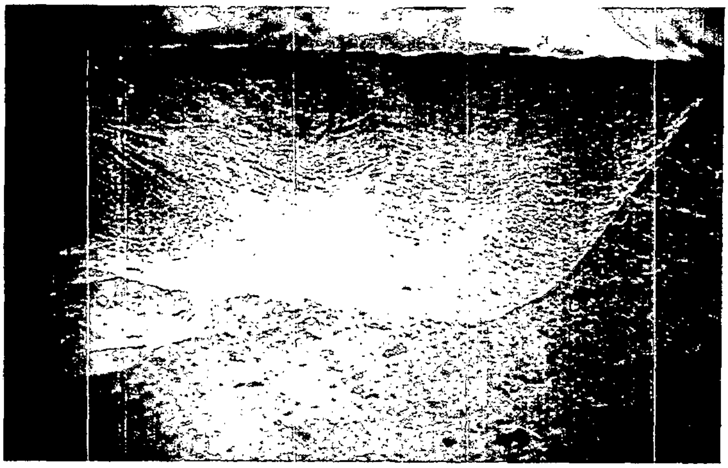
Fig 4 : Optical photograph showing detail of the skin/core microstructure which was observed in the part machined disc springs.



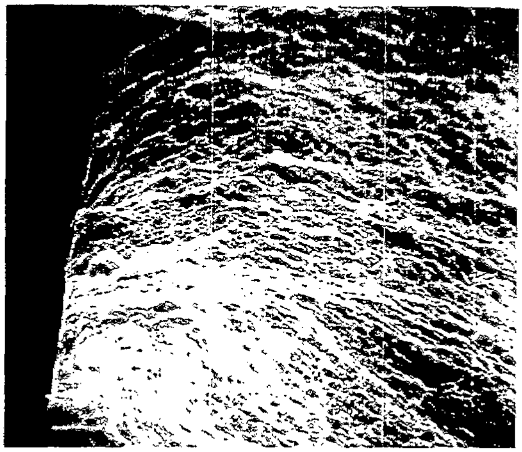
x 400

x 800

Fig 5 : Scanning electron photograph showing in further detail the skin/core microstructure of a part machined disc spring.



x 160



x400

Fig 6 : Scanning electron photograph showing the fracture surface of an AHZ coil spring. The failure is characteristic of fatigue.