

# Residual Stress Measurements of Hot Isostatically Pressed Silicon Nitride Rolling Elements

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**Abstract:** Ceramic materials used in rolling element bearing applications show some practical advantages over traditional bearing steels. Silicon nitride has been found to have a combination of properties suitable for certain high speed, low mass and high stiffness applications. The measurement of residual stresses is an important aspect of surface engineering to assist in correct design specification. An X-ray method was used since it is the only practical and non-destructible means of measuring residual stress of these materials at the pre-test and post-test stages. A pre-test survey of test sample residual stress within hot isostatically pressed silicon nitride supplied from selected manufacturers is presented. Experimental testing, surface examinations and residual stress measurements are described for case study silicon nitride failures. The role of the residual stress before and after fatigue testing is discussed. A modified four ball machine is employed to produce the accelerated rolling contact fatigue failures. © 1998 Elsevier Science Limited and Techna S.r.l. All rights reserved

## NOTATION

$d$	Interplaner spacing in the crystal
$E$	Elasticity modulus
FWHM	Full Width of Half Maximum
HIP	Hot Isostatically Pressed
$n$	Positive integral number indicating the order of diffraction
$\epsilon$	Quantity of strains
$\theta$	Diffraction angle
$\theta_0$	Diffraction angle in a stress-free condition
$\lambda$	X-ray wavelength
$\nu$	Poisson's ratio
$\Psi$	Angle between sample normal and diffraction plane normal

## 1 INTRODUCTION

An understanding of residual stress within complex materials is needed to evaluate the affect of the

manufacturing process, characterise failure modes and evaluate the in-service loading conditions of concentrated rolling contacts. It is important to assess the residual stress levels caused by both the manufacturing process and during the in-service conditions of rolling contact to enable correct design decisions to be made in respect to e.g. fatigue strength. The residual stress values within silicon nitride will vary due to primary processing (hot isostatically pressing) and surface finishing methods. In respect to surface finishing methods there is significant pressure to accelerate material removal rates to reduce product financial costs. In addition to traditional grinding and lapping methods new processes such as magnetic force (Kato and Ume-hara)<sup>1</sup> assisted and tribochemically (Jisheng *et al.*)<sup>2</sup> assisted grinding have been considered for silicon nitride materials. It is therefore necessary to monitor manufacturing residual stresses within the finished silicon nitride rolling elements. In-service residual stress known as “shakedown” is described in Johnson.<sup>3</sup> During the first repeated rolling, a process of plastic deformation leads to a steady

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state elastic deformation. The maximum at limit which shakedown occurs is known as the "shakedown limit". Although shakedown effect does influence the fatigue strength of metallic rolling contacts the influence on silicon nitride will be limited. It is acknowledged that residual stresses during rolling contact may be beneficial in that the fatigue strength is enhanced due to material shakedown.

Residual stresses were measured on steel rolling element bearings by Munro *et al.*<sup>4</sup> In that study the compressive residual stresses were shown to peak at the position of maximum shear stress caused by the normal contact pressure. Also, early fatigue failure was observed when residual compressive stress appeared near the surface. The residual stress measured by X-ray diffraction of silicon nitride rolling contact fatigue failures was performed by Hadfield *et al.*<sup>5</sup> Variation of measured residual stress with depth of delamination fatigue and brittle fracture was discussed. During that study the compressive residual stress values increased with silicon nitride delamination failure depth.

In this study experimental residual stress measurements of silicon nitride are presented. A feature of this study is the small irradiation area of 0.25 mm<sup>2</sup> that has enabled measurements of specific locations within the rolling contact path. Residual stresses caused by manufacturing and subsequent testing are assessed separately. The accelerated rolling contact fatigue tests using a modified four-ball machine are briefly outlined and referenced. Residual stress analysis by X-ray diffraction of the pre-test, contact path and failed rolling elements are presented. A brief description of the X-ray diffraction residual stress measurement methodology is presented. The results of the residual stress measurements are discussed with respect to contact conditions.

## 2 ACCELERATED ROLLING CONTACT FATIGUE TESTS

An accelerated rolling contact fatigue bench test was used to study the lubricated failures and in-service behaviour of silicon nitride. The tests were performed using a modified four-ball machine which are described by Hadfield.<sup>6</sup> This machine consists of an assembly which simulates the loads and motions within an angular rolling element bearing. A stationary steel cup represents a bearing outer-race, three lower balls represent the rolling elements within a bearing-race and the upper ball represents the inner-race. High contact stresses are produced due to the non-conforming contact between the upper-ball and three lower balls. The

assembly is loaded via a piston below the steel cup, from a lever-arm load. The upper-ball is assembled to a drive shaft via a collet and contacts with three lower-balls when the machine is stationary. The contacting positions between the upper ball and lower balls were immersed with lubricating oil. The oil lubrication is designated (LV) for low viscosity oil and (HV) for high viscosity oil. The low viscosity lubricant is a synthetic oil which has a kinematic viscosity of 12.5 c.s.°C at 40°C and 3.2 c.s.°C at 100°C and the high viscosity lubricant is a mineral oil of 200 c.s.°C at 40°C and 40 c.s.°C at 100°C. The silicon nitride balls were manufactured by a hot isostatically pressed (HIP) method. Average roughness (Ra) of the silicon nitride ball surfaces were 0.01 µm. The steel balls were grade 10 (ISO 3290-1975) carbon chromium steel with a surface roughness of 0.02 µm Ra. Lubrication film thickness was calculated using elastohydrodynamic theory for thin films. Shaft/upper-ball speed is set at speeds of 10 000 for ceramic/ceramic tests and 5000 r min<sup>-1</sup> for ceramic/steel tests. At these conditions, thin film separation exists and stresses may be considered Hertzian. Table 1 describes the basic test conditions for test "A" to "D".

Silicon nitride Vs silicon nitride tests "A", "B" and "C" were suspended without failure after 150 million stress cycles to the upper ball. The maximum contact pressures for tests A, B and C were 7.1, 7.6 and 8.1 GPa, respectively. The tests were conducted at 10 000 r min<sup>-1</sup> with the contact area immersed with the HV lubricant. Test "D" configuration was a steel upper contacting with three silicon nitride lower balls, this configuration simulates a hybrid rolling element bearing. A hybrid rolling element bearing being defined as bearing steel inner and outer races with silicon nitride rolling elements. In this case the silicon nitride surface was damaged by radial and lateral cracks propagated using Vickers (5 kg) hardness indenter. The lower balls failed after 0.54 million test cycles, full test results and surface analysis of the complete pre-cracked test series are reported by Hadfield *et al.*<sup>6</sup>

## 3 RESIDUAL STRESS MEASUREMENT

### 3.1 Background

The X-ray stress measurement was used to evaluate residual stress within the silicon nitride rolling element areas. A  $\sin^2\Psi$  method was used; this technique is appropriate for materials composed of a crystal structure. A detailed description of this method may be found from Farrahi *et al.*<sup>7</sup> A

Table 1. Fatigue tests "A" to "D"

Test	A	B	C	D
Contact (top ball on bottom balls)	Silicon nitride on silicon nitride	Silicon nitride on silicon nitride	Silicon nitride on silicon nitride	Steel on silicon nitride
Po (GPa)	7.1	7.6	8.1	6.4
Contact radius (mm)	0.21	0.22	0.23	0.24
Upper ball stress cycles	Suspend after 150 million	Suspend after 150 million	Suspend after 150 million	0.54 million
Oil	HV	HV	HV	LV

schematic of the measurement apparatus is shown as Fig. 1. When a stress is applied to a material, the interatomic distance within the crystal will be extended or compressed in proportion to its force within the elastic limit. The X-ray diffraction technique measures the variations of the interplaner spacing within the crystal and stress is then calculated. Using the Bragg's condition for diffraction (1), strain measured from the quantity of variations of the X-ray diffraction angle:

$$n\lambda = 2d \sin \theta \quad (1)$$

$$\epsilon = \frac{\Delta d}{d} = \cot \theta_0 \Delta \theta \quad (2)$$

Stress is then calculated from eqn (3):

$$\sigma = \frac{E}{1 + \nu} \frac{\partial(\epsilon)}{\partial(\sin^2 \Psi)} = -\frac{E \cot \theta_0}{2(1 + \nu)} \frac{\partial(\theta)}{\partial(\sin^2 \Psi)} \quad (3)$$

Equation (3) is described by a constant "K" and variable "M" shown as eqn (4):

$$\sigma = KM \quad (4)$$

where

$$K = -\frac{E \cot \theta_0}{2(1 + \nu)} \quad (5)$$

and

$$M = \frac{\partial(\theta)}{\partial(\sin^2 \Psi)} \quad (6)$$

The value of "K" is called the "X-ray elastic constant" and has already been measured on many materials. The variable "M" is the gradient of a  $\sin^2 \Psi - 2\theta$  diagram and express strain of a measured material. The "M" value is measured by peak shift of the diffraction profile found from each  $\Psi$  angle. There are several methods to decide the diffraction peak position. The full width of half intensity (FWHM) method is commonly used when residual stresses of metallic materials are measured. In the case of silicon nitride materials usually there are many diffraction planes, therefore, sometimes it is difficult to identify background of diffracted profile of X-ray because two or more peaks are close to each other. In that case, the FWHM method is not available, but peak top method is usable. In the peak top method, a peak profile is approximated by a quadratic equation and the centre of the peak is decided. In the present paper, all of the measurements were performed using the peak top method. An example of the measurement is shown in Fig. 2. The X-ray elastic constant of silicon nitride is taken as  $-1280 \text{ MPa degree}^{-1}$  and the gradient M is found to be  $0.0869^\circ$ . The calculated value of  $-111$  residual stress is thus the product of "M" and "K".

### 3.2 Measuring procedure

The incident X-ray was varied at 0, 15, 25, 35 and  $45^\circ$  and these are called the angle between the sample normal and diffraction plane normal ( $\Psi$  angle). The measurements were performed on a Rigaku Strainflex MSF-2M machine. The X-ray tube has a maximum load of 40 kV and 40 mA, the incident beam is radiated through a collimator

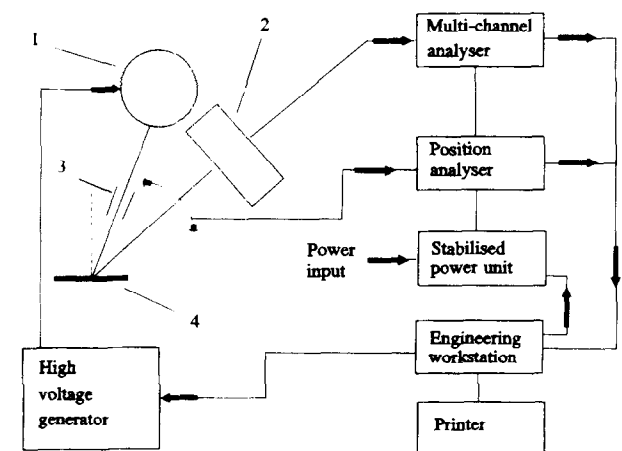


Fig. 1. Schematic diagram of X-ray apparatus. (1) X-ray tube (rotary type); (2) PSPC (position sensitive proportional counter); (3) collimator; (4) specimen.

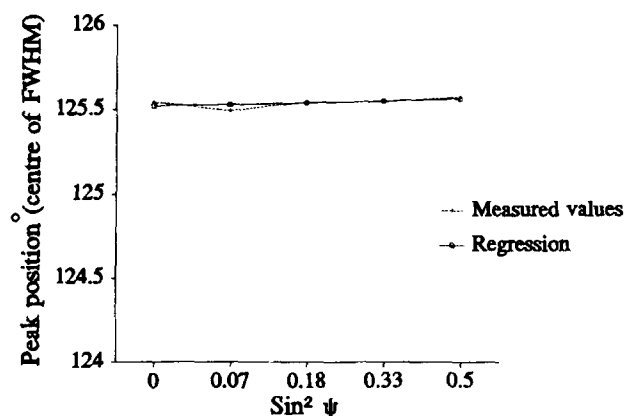


Fig. 2. Typical X-ray diffraction stress gradient.

which has a diameter of 0.5 mm. Diffracted X-ray is detected by a position sensitive proportional counter (PSPC). The intensity of diffracted X-ray is changed to voltage and analysed by a multi-channel analyser. The X-ray tube is selected, for silicon nitride materials Cr-K $\alpha$  is used typically and vanadium filter is combined with Cr-K $\alpha$ . The characteristic X-ray influences penetration depth and diffraction plane. Measurement parameters are described in Table 2.

The choice of measurement parameters is complex and based upon the diffraction plane and X-ray characteristics. The measured X-ray diffraction peak from a particular diffraction plane should have diffraction peak close to 180°, and also be high intensity and independent. A chromium target was chosen as most diffraction angles were closer to 180° than, for example, iron, cobalt, nickel, copper, molybdenum or silver targets. The choice of alternative targets can however be useful for approximating stress gradients in silicon nitride due to the variety of measurement depths. A (411) diffraction plane was chosen which has an intensity of 18 and diffraction angle of 125.4°. Other competing diffraction planes were considered i.e. (321) and (212) which have intensities of 117.2° and 131.5° with intensities of 39 and 16, respectively. The (212) plane is potentially suitable but a peak from the (330) plane is too close (129.1°) and is probably related to other crystalline phases such as SiNO.

Table 2. X-ray stress measurement parameters

Characteristic X-ray	Cr-K $\alpha$
Diffraction plane	(411)
Diffraction angle	125.4°
Irradiation area (mm <sup>2</sup> )	0.25
Intensity	18
$\lambda$ (Å)	2.29
Measurement time (s)	900

## 4 RESULTS AND DISCUSSION OF RESIDUAL STRESS MEASUREMENTS

A measurement survey of residual stresses within procured HIP silicon nitride 12.7 mm diameter balls was conducted. Seven different manufacturers were considered to determine the range of residual stress which could exist within a typical material. In addition the survey may be used as a residual stress guide for consideration by mechanical designers. Variations of residual stress exist due to the variation of additives, processing and finishing method amongst the manufactures. Table 3 shows the residual stress values and measurement accuracy for each ball supplied by manufacturers "A" to "G". The results of the residual stress survey are placed in ascending numerical order, Table 3. All pre-test measured values were found to be compressive and ranged from 54 MPa to 111 MPa and averaged 75 MPa. The exact nature of the material processing and surface finishing methods are unknown and as such the nature of variation of the measured residual stress values is not expressed. The values are reasonable considering the material pressing pressures of 300 MPa maximum, thermal expansion from extremely high temperatures and surface finishing operations.

The position and direction of residual stress measurements in test "B" upper ball were conducted in detail to assess possible variations according to the in-service stress field. Figure 3(a) illustrates the positions, magnitude and measurement direction of the measurements in relation to the wear path. The measurements were positioned at the contact edges and contact centre. The maximum Hertz stresses for this contact are 7.6 GPa maximum compressive located at the wear track centre, 1.0 GPa maximum tensile located at the contact edges with a alternating maximum shear stress of 2.4 GPa located 111.0  $\mu$ m below the surface. The results indicate that compressive residual stress has increased at position "B" in the x-direction and position "A" in the y-direction. The residual stress has decreased at position "A" x-direction. The differences of residual stress measurements can be explained in terms of the stress

Table 3. Survey of residual stress

Manufacturer	Residual stress (MPa)	Accuracy (MPa)
A	-54	+/-19
B	-55	+/-26
C	-69	+/-32
D	-70	+/-28
E	-74	+/-8
F	-94	+/-29
G	-111	+/-40

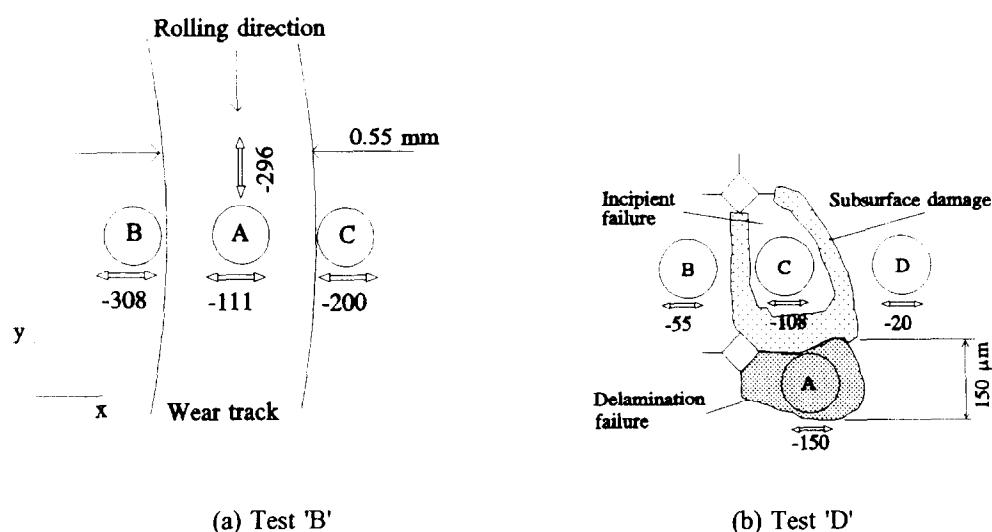


Fig. 3. Residual stress of silicon nitride rolling elements.

field and rolling direction although further experimental investigations are required to confirm the directionality of residual stress. Plasticity or shakedown effects may also have a considerable influence on the measured residual stress field within the material although it is not measured at the maxima. This result does show the importance of measurement direction when interpreting X-ray residual stress measurements in tribological applications.

A failed lower silicon nitride ball which contained surface pre-cracks was measured at specific areas. Figure 3(b) shows the measurements in relation to the failed area. The failed area is complex and has been examined using acoustic microscopy.<sup>6</sup> The failure area contains a delamination failure, subsurface crack propagation (measured using an acoustic microscope) and an unfailed incipient failure region. Measurement 'A' within the delamination area is 150 MPa compressive. The delamination depth measured using laser

microscope is shown as Fig. 4. The failure depth varies and is maximum at 20  $\mu\text{m}$ . The effective measurement depth is 195  $\mu\text{m}$  within the material. The variation of residual stress with depth of delamination failure was discussed previously.<sup>5</sup> This study described much higher compressive stress within delamination fatigue failures at corresponding failure depths. The difference of the present delamination measurement could be a result of lower contact stresses and steel Vs silicon nitride contact. The residual stress value at point "C" is 108 MPa compressive. If the maximum residual stress occurred at the point of maximum orthogonal shear stress then the difference between point "A" is understandable. At position "B" and "D" (Fig. 3(b)) the measurements are substantially lower than those presented for silicon nitride vs silicon nitride contact. This could be a result of the subsurface crack propagation releasing HIP treatment compressive residual stress. It is clear that

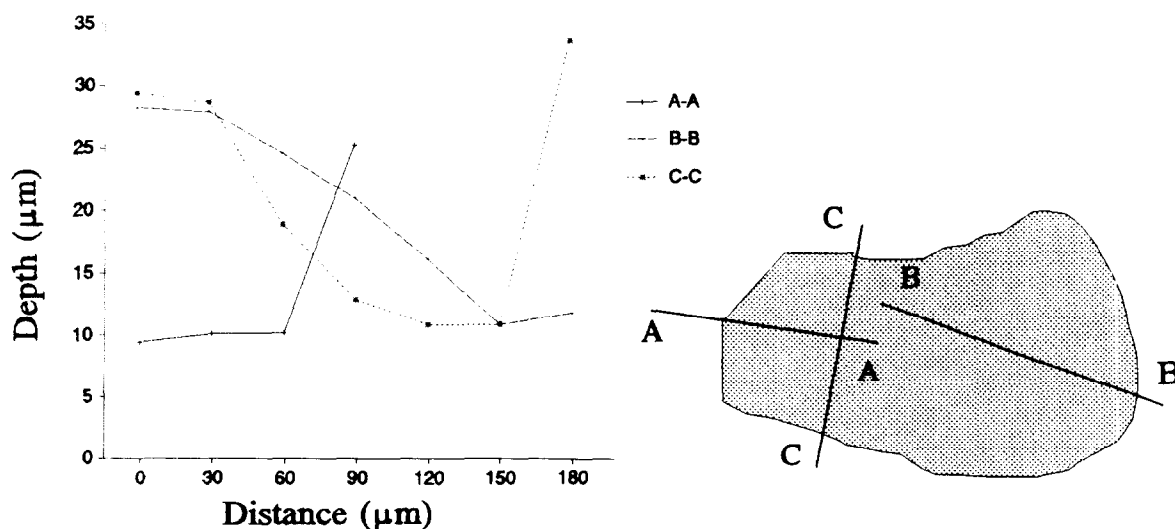


Fig. 4. Delamination depth profiles.

more detailed research programmes are required to investigate the nature of residual stress and its effect on lubricated silicon nitride rolling contact fatigue.

## 5 CONCLUSIONS

X-ray measurement directionality is important to consider when interpreting residual stress values. All residual stress values in the pre-test and post-test condition were found to be compressive. The average residual stress amongst seven manufactures was 75 MPa compressive. A small irradiation area of 0.25 mm<sup>2</sup> has enabled measurements of specific locations within the rolling contact path and adjacent locations. The present measurements indicate that residual stress decreases during rolling contact fatigue tests on HIP silicon nitride.

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