

## Characterization of Mechanical Reliability of Silicon Nitride Microturbine Rotors

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### Abstract

This paper summarizes the recent results on component characterization efforts carried out to verify the mechanical reliability of SN237 and SN281 silicon nitride microturbine rotors manufactured by Kyocera. Mechanical properties of biaxial discs machined from airfoils of microturbine rotors were evaluated by a ball-on-ring test technique. Results showed that the mechanical properties of samples from airfoils with as-processed surfaces exhibited lower characteristic strength than those machined from the hub region with as-machined surfaces. The differences in mechanical performance and reliability between as-processed components and simple-shaped test coupons appear to arise mainly from differences in strength limiting flaw type and population.

### I. Introduction

The Advanced Microturbine Program was initiated in 2000 by the U.S. Department of Energy to improve the energy efficiency (> 40%) and reduce the NO<sub>x</sub> emission (< 7 ppm) of the next generation microturbine systems [1]. These systems would provide a viable distributed energy resource for commercial, industrial, and institution sectors. Due to their superior thermomechanical performance and oxidation resistance at elevated temperatures, advanced silicon nitride ceramics with elongated reinforcing grains have been considered for hot-section components in advanced microturbines to achieve the stated goals. Therefore, several microturbine companies have been funded by the DOE Ceramic Microturbine Program to undertake probabilistic component designs and life predictions to achieve successful implementation of the ceramic components, i.e., turbine rotor, vane, and combustor liner, for advanced microturbine systems. Previous studies have demonstrated that the mechanical properties of advanced silicon nitride ceramics generated from simple-shaped test coupons machined from production billets can over-estimate the long-term mechanical reliability and lifetimes of real components [2]. Thus, it is critical to develop a database for actual components with complex-shaped geometry for verifications of probabilistic component design and life prediction subjected to application conditions.

Both Ingersoll-Rand Energy System (IRES) and United Technology Research Center (UTRC) were awarded contracts by DOE for the Ceramic Microturbine Program in 2000 to increase the experience base concerning the integrated system design with ceramic components and the behavior of silicon nitride components in microturbine environments. The ceramic microturbine plan of IRES was to develop a low risk path to demonstrate the feasibility of silicon nitride microturbine rotors that could be operated within the proven limit of IRES existing technology [3]. The low risk path developed by IRES built upon the proven technology developed by Kyocera

for its silicon nitride high volume turbocharger manufacturing processes. The microturbine system, selected for ceramic component demonstration, was the IRES 70 kWe PowerWorks™, which is a co-generation system.

UTRC has been working with Pratt & Whitney Canada (P&WC) to develop and produce a 400 kW microturbine system (i.e., ST5+) based on P&WC's PW207 helicopter engine modified to meet the needs of industrial power generation [4]. The introduction of silicon nitride components (vane ring and integrally bladed rotor) has a great potential to improve the electrical efficiency from 30% to 40% and reduce the NOx emission to < 7 ppm via the higher turbine inlet temperature (TIT) of 1150°C compared to the baseline metallic engine.

This paper describes the results of a recent component verification effort involving as-sintered silicon nitride microturbine rotors. The as-processed surface strength was measured using a miniature biaxial specimen, which was prepared by diamond core drilling. Both optical and scanning electron microscopy techniques were used to elucidate the factors limiting the mechanical performance and reliability.

## II. Experimental Procedures

The microturbine rotors examined in this study were manufactured from SN237 and SN281 silicon nitride by Kyocera Industrial Ceramic Corp, US, and Kyocera Automotive Division, Kagoshima, Japan, respectively. The SN237 silicon nitride was developed for application limit < 1200°C, which is applicable to the IRES 70 kWe PowerWorks™ microturbine system with designed TIT of ~ 1000°C [3]. The SN281 was developed for the Japan Hybrid Gas Turbine program with TIT of 1250°C [5], and the TIT for ST5+ is ~ 1150°C [4]. Figure 1 shows the photos of the as-received SN237 and SN282 silicon nitride rotors prior to specimen preparations. Biaxial discs were machined from both the airfoil and hub surfaces by first diamond core drilling small cylinders having a nominal diameter of 6.0 mm. Each cylinder was then machined on the back face only until the thickness was 0.4 to 0.5 mm. In this way one face of each specimen always consisted of the as-processed surface of the airfoil. During testing, the as-processed airfoil surface was loaded in tension, while discs from hub region were tested such that the 600 grit machined surfaces were in tension.

The biaxial flexure strength [6-8] was measured using a ball-on-ring arrangement. The test fixture consisted of a 1 mm diameter WC ball, which was mounted to a miniature load cell. The lower support 5 mm diameter ring was fabricated from a high-strength polymer. The test fixture was mounted on a stage driver on a vertical stepper motor (Z stage), which was affixed to the X-Y stage for positioning in the horizontal plane. A personal computer controlled all three stages. After placing a specimen on the lower support ring, the X-Y stages were used to position the assembly directly under the upper load ball. The specimen was loaded to failure at a displacement rate of 0.05 mm/s. The computer monitored and recorded the displacement, load, and time.

The strength,  $S_b$ , was calculated from the equation

$$S_b = 3P(1+\nu)/(4vt^2) \cdot [1 + 2\ln(a/b) + ((1-\nu)/(1+\nu))(1 - b^2/2a^2)(a^2/R^2)] \quad (1)$$

where P is the ultimate sustained load, a is the radius of the support ring, b is the effective radius of contact of the loading ball on the specimen, R is the specimen radius, t is the specimen thickness, and  $\nu$  is Poisson's ratio [6]. As a first approximation, b was taken as  $t/3$ .

Data generated for bend bars machined from both SN237 and 281 silicon nitride production billets were also included as a baseline for reference. In the case of SN281 rotor bend bars were also machined from the hub region and the characteristic strength generated will be compared with those converted from biaxial data. These bend bars were all longitudinally machined per ASTM C1161-2002 standard with 600 grit surface finish [9], and were tested at 20°C and 30 MPa/s per ASTM C1368 standard [10]. The accumulated strength data were then further analyzed. The strengths for each test set were fit to a two-parameter Weibull distribution using the program CERAMIC [11], which uses maximum likelihood estimation advocated in ASTM C1239 [12]. Reported results are uncensored because fractography analysis was only conducted to identify strength-limiting flaws for limited number of samples via optical and scanning electronic microscopy. Both optical and scanning electron microscopy techniques were carried out to determine the strength limiting flaws in the biaxial flexure disks sectioned from the airfoil and hub regions.

### III. Results and Discussions

Results of uncensored Weibull strength distribution obtained for samples machined from airfoil and hub region of IRES SN237 silicon nitride microturbine rotor are shown in Table 1, and Fig. 2 and 3. Note that the airfoil and hub samples are tested with the as-processed and machined surface in tension, respectively. Mechanical results show that discs with as-processed surface from airfoils exhibit characteristic strengths that are ~22% lower than those obtained from hub region with machined surface. However, samples from airfoils regions exhibit a higher Weibull modulus than the value obtained for samples from hub region. On the other hand, the machined hub discs exhibited comparable characteristic strength and Weibull modules to those obtained from bend bars machined from production billets (Table 1). The strength value measured for the machined biaxial discs in general is approximately 30-40% higher than that obtained using ASTM C1161B standard machined test bend bars due to its smaller load factor as compared with that for standard bend bars [13].

Mechanical results of airfoil and hub samples machined from UTRC SN281 silicon nitride microturbine rotor are summarized in Table 1, and Figs. 4 and 5. Results show that the characteristic strength of as-processed airfoil samples is ~39% lower than that obtained for the machined hub samples, similar to the results obtained from SN237 rotor. Also, both airfoil and hub samples exhibit similar Weibull strength distribution in spite of the distinct difference in surface condition. The conversion of biaxial strength to flexure strength using the results of machined hub biaxial discs [14] yields a flexure strength of 585 MPa, which is comparable to the characteristic strength (540 MPa) obtained for the machined standard test bend bars from the same hub region (as shown in Table 1). Previous study on alumina ceramic has also shown that biaxial strength can reliably predict the flexure strength, which is consistent to the actual value measured for bend bars machined from the same billets [15]. Nonetheless, the flexure strength obtained for the machined bend bars from the SN281 rotor is still 17% lower than that obtained for the machined bend bars from production billets, probably due to the differences in microstructure and chemical composition between complex-shaped components and simple-shaped production billets.

Following the biaxial strength evaluations, scanning electron microscopy (SEM) analysis was carried out to determine the strength limiting flaws in samples machined from the airfoil and hub region. SEM examinations of fracture surfaces of selected SN237 biaxial discs show that the typical strength limiting flaws observed in airfoil samples originate from the surface irregularities, i.e., large concave areas, arising from the component forming process (Fig. 6a). For the machined hub samples the strength limiting flaws are in general

associated with the long machining defects (~ 50-80  $\mu\text{m}$  long and 2-3  $\mu\text{m}$  deep). On the other hand, the typical flaws observed in SN281 rotor airfoil are large pores that were not eliminated during the sintering process. As for the machined SN281 rotor hub samples the strength limiting flaw was associated with the very large elongated grains (~100-150  $\mu\text{m}$  long and 10-15  $\mu\text{m}$  diameter). The presence of these large elongated grains is very typical for SN282 silicon nitride due to its high sintering temperature.

Results of mechanical testing and SEM examinations for both SN237 and 281 microturbine rotors showed that the mechanical properties generated from airfoils with as-processed surfaces are inferior to those from hub region with machined surface due to the difference in type of strength limiting flaw. However, mechanical results showed that using the strength value generated for the machined biaxial discs from the hub region can reliably predict the flexure strength, as demonstrated by the actual strength value measured for the machined bend bars from the same component region. Note that the measured flexure strength for rotor component is still inferior to the value obtained for bend bars machined from production billets due to significant differences in microstructure and chemical composition.

The present study suggests that using the mechanical data generated from machined simple shape test samples could overestimate the lifetime of components due to the significant difference in mechanical performance and reliability. Therefore, one has to exercise caution when using the database from simple shaped test coupons to predict the probabilistic lifetime of component under the application conditions. It is critical to re-employ the database generated from the complex shaped components for verification of probabilistic component design and life prediction to ensure long-term mechanical reliability and lifetime under application conditions.

#### IV. Summary

Biaxial mechanical tests were carried out on discs machined from SN237 and SN281 silicon nitride microturbine rotor for database generation. Results show that the samples from as-processed airfoil region exhibit characteristic strength that is significantly lower (20-40%) than that obtained from machined hub. Also, the strength of the machined biaxial discs is much inferior to the value obtained from standard test bend bar due to the large difference in surface-to-volume ratio. However, the biaxial strength obtained for the machined discs can reliably predict the flexure strength for the bend bars machined from the same component. On the other hand, SEM examinations reveal significant change in the type of strength limiting flaw between as-processed and machined sample, resulting in different mechanical performance and reliability. Thus, a key lesson learned in this work is that mechanical properties of complex-shaped ceramic components are often quite different from those determined from standardized simple-shaped test specimens. The application of component characterization can address this limitation by providing a mechanical database directly from the ceramic components in question for probabilistic component design and life prediction.

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Table 1. Summary of uncensored Weibull and strength distributions for Kyocera SN2337 and 281 silicon nitride biaxial discs machined from microturbine rotor. Data of SN237 and 281 silicon nitride production billets longitudinally machined per ASTM C1161 are also included for reference.

Material	# of Spmns. Tested	Stressing Rate (MPa/s)	Temp. (°C)	± 95% Uncens.		± 95% Uncens.	
				Weibull Modulus	Weibull Modulus	Chrctstic Strength (MPa)	Chrctstic Strength (MPa)
SN237 Rotor APAF*	13	01. mm/s	20	19.13	11.83, 28.26	814	788, 840
SN237 Rotor AMHB**	13	0.1 mm/s	20	9.74	5.79, 14.10	1037	969, 1105
SN237 Billet	15	30	20	13.99	8.94, 19.98	979	939, 1019
SN281 Rotor APAF*	26	0.1 mm/s	20	14.42	10.46, 19.02	548	532, 564
SN281 Rotor AMHB**	20	0.1 mm/s	20	14.36	9.80, 19.87	898	867, 928
SN281 Billet	15	30	20	17.26	11.05, 25.04	702	687, 725

\*Specimens were machined from airfoil region with as-processed surface (APAF).

\*\* Specimens were machined from hub region with as-machined surface (AMHB).

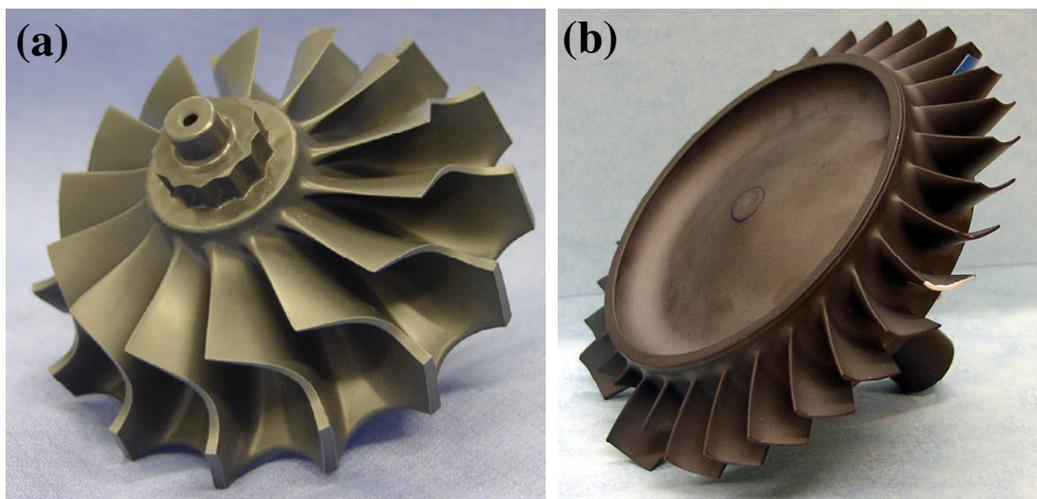


Figure 1. Photos show the Kyocera SN237 (a) and SN281 (b) silicon nitride microturbine rotor designed by Ingersoll-Rand Energy System and UTRC, respectively.

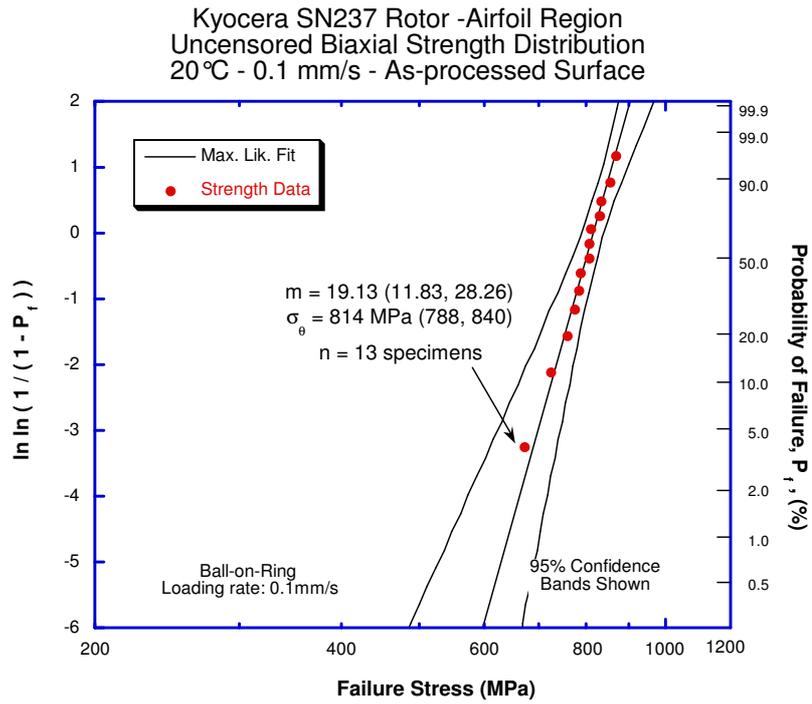


Figure 2. Uncensored biaxial strength distribution for SN237 rotor airfoil samples with as-processed surface at 20°C.

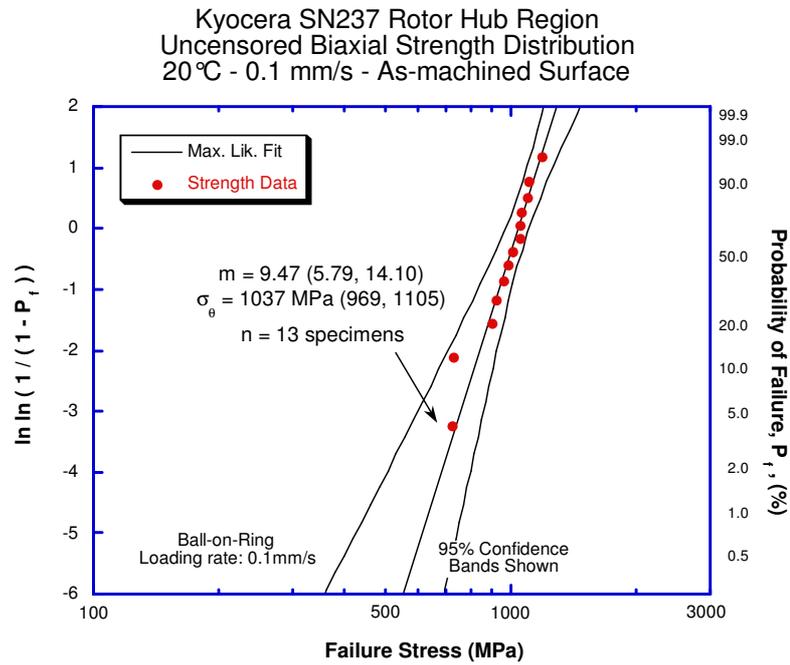


Figure 3. Uncensored biaxial strength distribution for SN237 rotor hub samples with machined surface at 20°C.

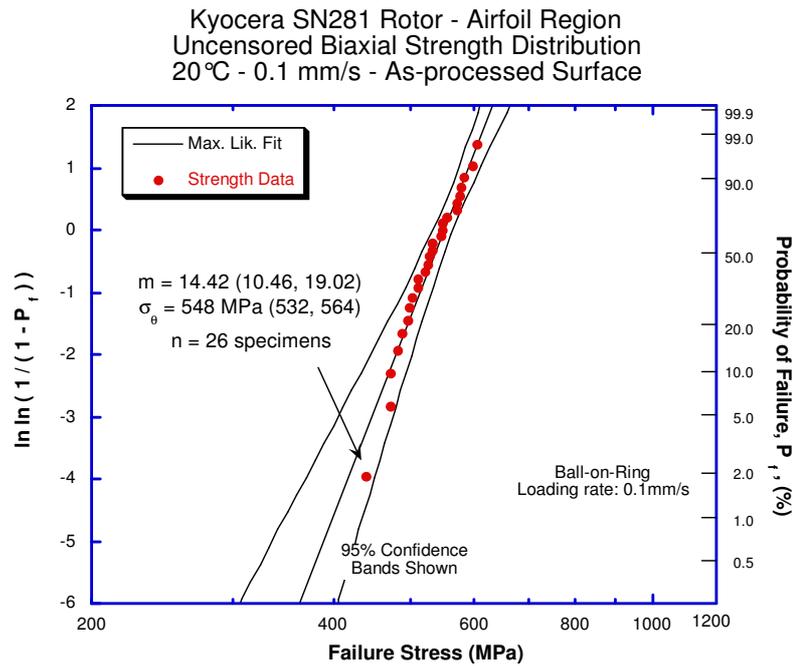


Figure 4. Uncensored biaxial strength distribution for SN282 rotor airfoil samples with as-processed surface at 20°C.

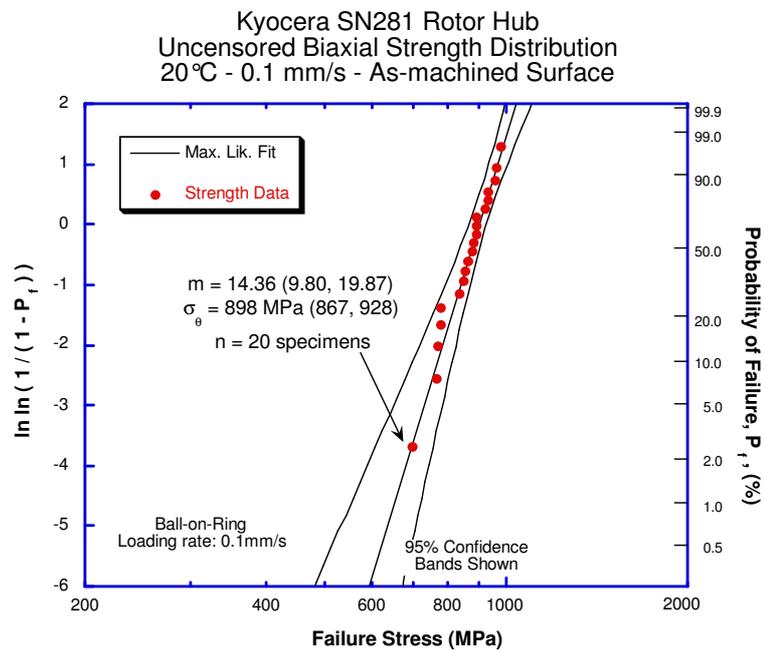


Figure 5. Uncensored biaxial strength distribution for SN282 rotor hub samples with machined surface at 20°C.

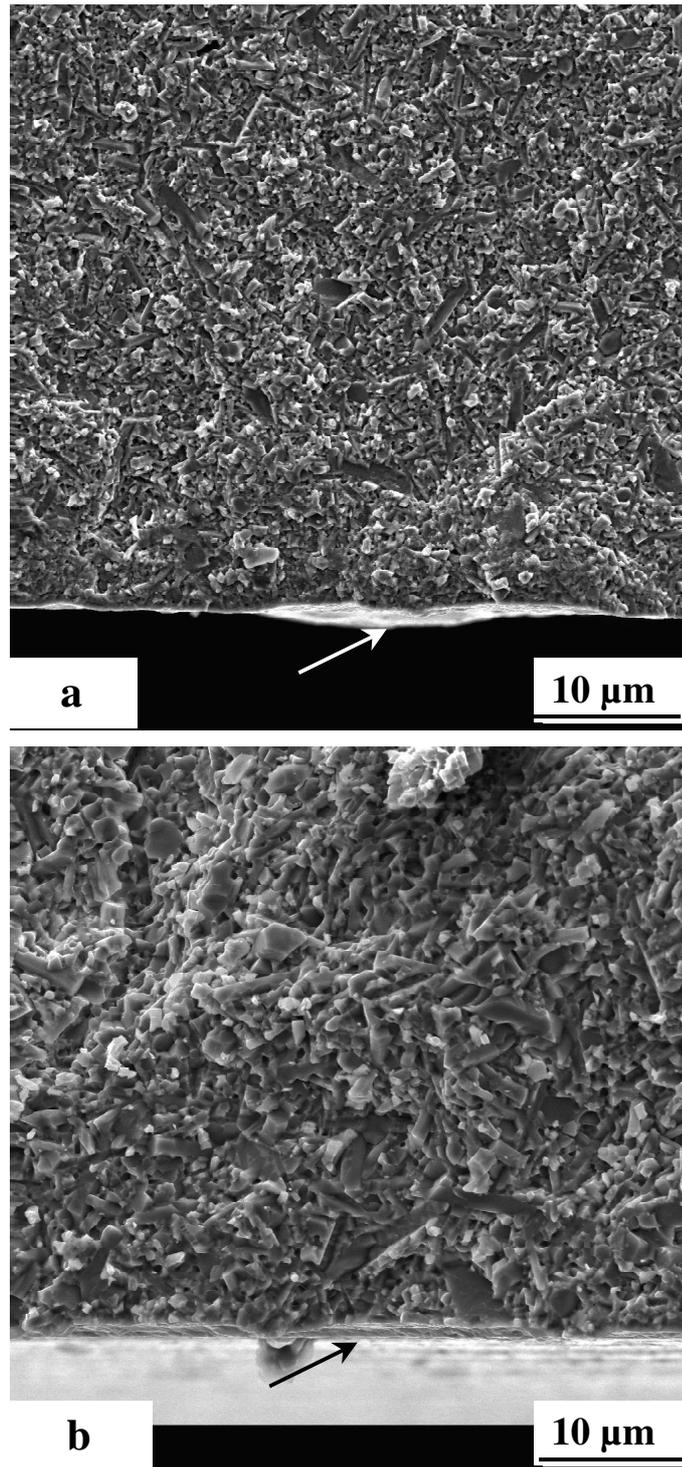


Figure 6. SEM micrographs of fracture surface of biaxial disc from (a) airfoil and (b) hub region. The arrow indicate the surface irregularity (a) and machining groove (b).

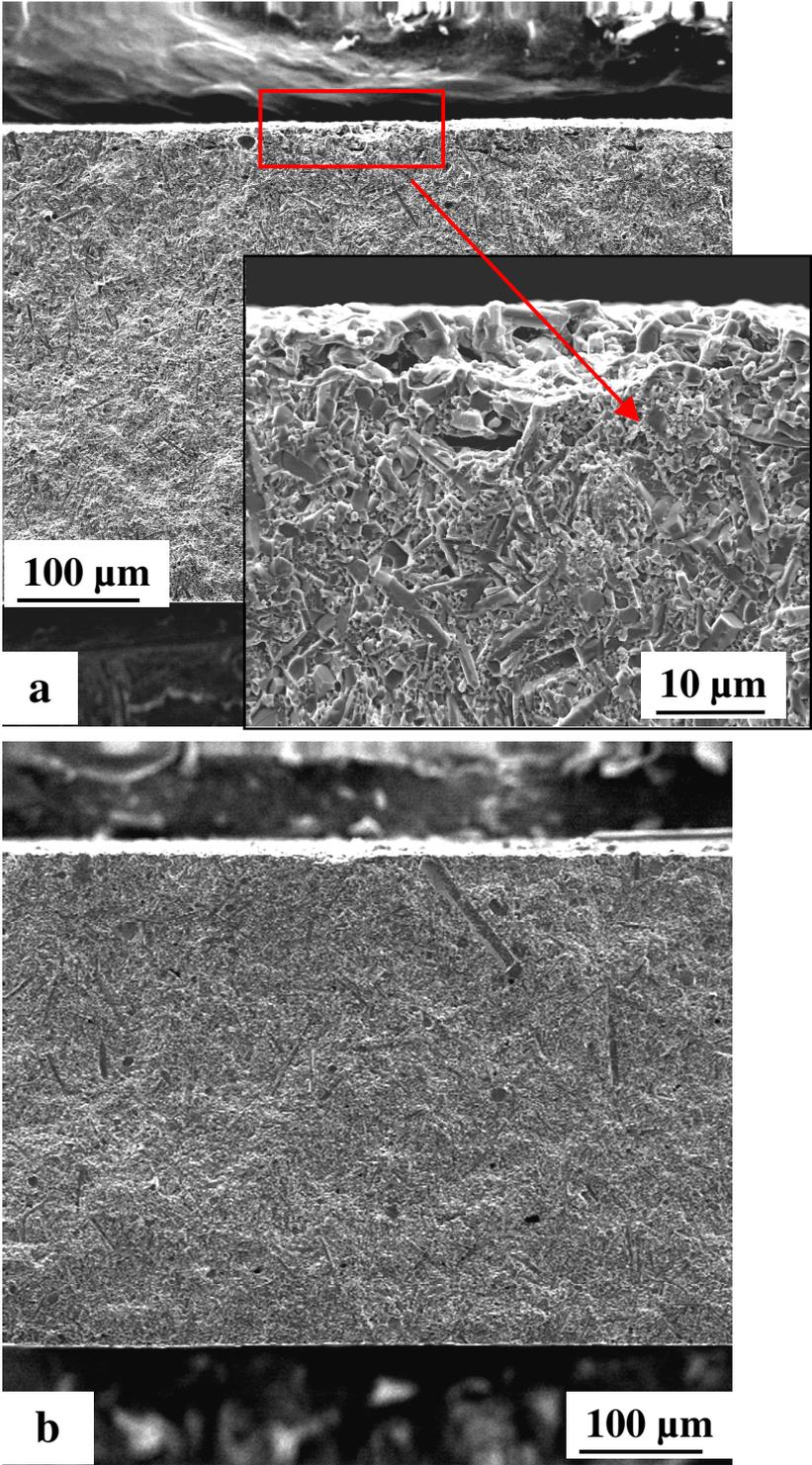


Figure 7. SEM micrographs of fracture surface of biaxial disc from (a) airfoil and (b) hub region. The insert in (a) shows a large pore present on the as-processed airfoil.