# Irradiated Single Crystals for High Temperature Measurements in Space Applications

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#### **ABSTRACT**

While spacecrafts experience temperatures from -120 to 110°C on the orbit, their surface reaches extremely high temperatures, well above 1000 °C, during descent into the atmosphere due to aerodynamic heating. Sophisticated insulation systems are designed for thermal protection. One of the steps in designing a protection system is experimental temperature measurements.

Neutron flux induces point defects formation and accumulation in diamond and SiC single crystals, which causes overall lattice expansion. During thermal annealing this process is reversed, so the annealing temperature and time result in the "reduced" lattice parameter (measured by X-Ray diffraction), which allows determining the maximum temperature, if the exposure time is known. This paper describes the use of irradiated single crystal high temperature sensors for measuring temperatures in thermal protection systems during spacecraft descent, as well as other space applications. These additional applications include measuring the furnace temperature during single crystal growth in space at zero gravity, and measuring the rocket combustion chamber turbo pump temperature.

### INTRODUCTION

New efficient engine and thermal protection systems design is the major tasks for the space industry. Engine efficiency increases with its operating temperature, which is limited by materials. New high-temperature materials are being developed, and include high-temperature alloys, composites, and ceramic materials. Since materials strength decreases with temperature, it is essential to develop adequate cooling systems. As the maximum operating temperature is normally just under a hundred degrees below the maximum allowed for a particular material, it is extremely important to gain knowledge of temperatures reached in operating devices. While predictive thermal models exist, the trusted temperature data can be obtained only experimentally. Accurate experimental temperature measurement techniques become especially useful during the engine fine tuning, although measuring high temperatures in operating engine is challenging, since:

- 1) the operating temperatures are high;
- 2) normally, large temperature gradients are present, and need to be accounted for;
- 3) the geometry of engine elements is complex;
- 4) new generation engine elements become smaller and smaller;
- 5) engine elements should stay intact during testing, i.e. the thermal sensor should not cause engine parts damage;
- 6) sensor should not affect tested material thermal properties;
- 7) getting readings from sensors installed in an operating engine is complicated.

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All experimental temperature measurements are based on some sort of properties or matter state change (i.e. fluid volume or pressure, electric force induced by two dissimilar materials contact, etc.). There are over 30 different phenomena and materials properties used for measuring temperature [1], with even a larger number of temperature measurement tools available today. There is a vast variety of applications, which necessitate a need for new methods, enhanced measurement range, accuracy, reduced sensor size, etc. Any new advantageous method allows building better engines and spacecrafts.

Measuring temperature in hard-to-access moving parts, such as rotating turbine disks, or jet nozzles, is even more challenging. These kinds of parts carry most of the mechanical, as well as thermal loads, which necessitates precise thermometry. In most cases, "conventional" thermometry techniques are not adequate for such applications. For example, signal from thermocouples needs to be somehow transferred from the measured moving parts to a readout unit. While these kinds of mechanisms exist, they cause additional errors due to contact friction, and are not usable at all at high rotating speeds. Moreover, they change the thermal transfer conditions, and the "true" part temperature can not be obtained.

Temperature measurement methods in hard-to-access parts can be divided into two categories based on their readout: contact and non-contact. The use of either one depends on the actual application. This paper describes the principle of operation, and space applications of a novel non-contact high temperature sensor based on the annealing of point defects in irradiated single crystals. The sensor was originally designed and manufactured by Kurchatov Institute in Moscow, Russia [2, 3].

# SENSOR DESCRIPTION

Single crystal (i.e. diamond or 3C-SiC) neutron irradiation causes point defects, vacancies and interstitials in the crystal lattice. The activation energy spectrum for these defects is continuous, ranging from fractions to several electron volts. Therefore, the defect annealing process (interstitial-vacancy annihilation) is continuous in time. For example, defects in 3C-SiC irradiated at 100 °C can be annealed continuously from 150 to 1400 °C. Point defects cause crystal lattice expansion (swelling), which increases with defect concentration. Irradiation-induced lattice swelling, easily measurable with X-Ray diffraction (lattice parameter measurement using Bragg's law), decreases with the anneal temperature and time.

Several thousand crystals will become high temperature sensors after irradiation, and further calibration. Calibration process consists of annealing irradiated crystals at known temperatures and different times, so that a calibration plot, similar to one show in Figure 1, could be created. Each sensor is then installed into a part being tested for a known period of time at an unknown maximum temperature. Defects in the sensor are partially annealed, causing a swelling reduction. Upon sensor extraction after testing, X-Ray diffraction analysis is performed. For the stationary heating regimes the change in the lattice parameter and the test time allows determining the maximum temperature that the extracted sensor has seen from the partial calibration plot (Figure 1a). As an example, for the part heated for 200 minutes, the resulted lattice expansion change of 1.8% was measured, which corresponds to 957 °C. Complicated heating profiles can also be accounted for if an approximate relative thermal time profile is known. In this case the correction in terms of the equivalent reduced time needs to be introduced (Figure 1a). For example, a tested part was exposed to T<sub>max</sub> for 10 minutes, then to 98% of T<sub>max</sub> for 20 minutes, and then to 96% of T<sub>max</sub> for 200 minutes, resulting in the total lattice expansion

of 1.75%. First, we approximately determine the upper bound of the maximum temperature at 1.75% lattice change achieved in 10 minutes, which corresponds to 1012 °C (Figure 1a).

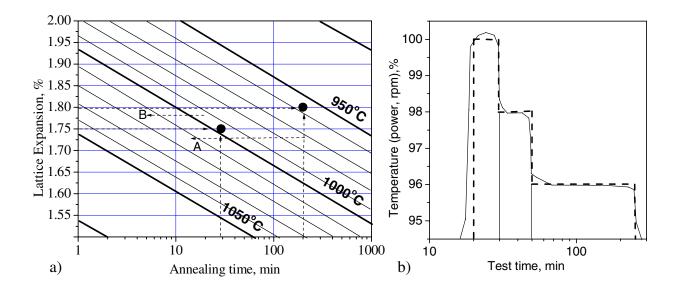


Figure 1. a) Partial calibration plot: irradiated 3C-SiC lattice expansion change dependence on the annealing time and temperature; b) Replacement of the continuous function with the step function.

This is really an upper bound, as the lower temperatures also contributed to lattice relaxation. Now, we can use the absolute temperatures instead of the relative temperatures: 10 minute exposure at 1012 °C, followed by 20 minute exposure at T=0.98T<sub>max</sub>=992 °C, followed by 200 minute exposure at T=0.96T<sub>max</sub>=972 °C. It's important to note that the lattice expansion decrease caused by 972 °C for 200 minutes is equivalent to 14 minute exposure at 1012 °C (see point A in Figure 1a), and similar, annealing at 992 °C for 20 minutes is equivalent to a 5 minute anneal at 1012 °C (see point B in Figure 1). Therefore, this particular non-stationary heating regime is equivalent to the maximum temperature anneal for 10+14+5=29 minutes. This would be an equivalent time of 29 minutes anneal at the maximum temperature as the non-stationary annealing gives the same result in terms of the crystal lattice expansion change. Now, the maximum temperature of 996 °C is determined based on the lattice parameter change of 1.75% and the equivalent time of 29 minutes (Figure 1).

In practice, temperature, power, rpm, and other parameters are continuous smooth functions of time in non-stationary regimes. These smooth curves are replaced with step functions graphically, and the time of each step is replaced with the equivalent time at maximum temperature as described above (Figure 1b). It should be noted that the equivalent time is always less than the actual testing time, but greater than the time of the maximum temperature exposure. Major 3C-SiC sensor characteristics are compiled in Table 1.

Table 1. 3C-SiC sensor characteristics.

Measured temperature range	150-1400 °C
Measurement error over the whole temperature range	±15 °C
Maximum temperature ramping speed	< 200 °C/sec
Sensor size	0.3-0.5 mm
Exposure time range	1-5000 minutes
Chemical stability in acids and bases	stable

# SENSOR SPACE APPLICATIONS

In some cases temperature gradient change can be measured by means of multi-crystal sensors. Such multi-sensors consist of a metal capsule 3 to 5 mm long, which contains several single crystals (Figure 2). After testing, the capsule is placed in the nitric acid solution for sensors extraction. Figure 2 shows schematics of a sensor shell positioned in the rocket nozzle, cooled with a fluid passing through special channels. The shell is 1 mm wide, 4 mm long, containing three 3C-SiC sensors. In this particular case, testing was performed in the lab environment, and theoretically, thermocouples could have been used. However, there were many test points, causing complications with hermetic sealing, so encapsulated sensors were employed.

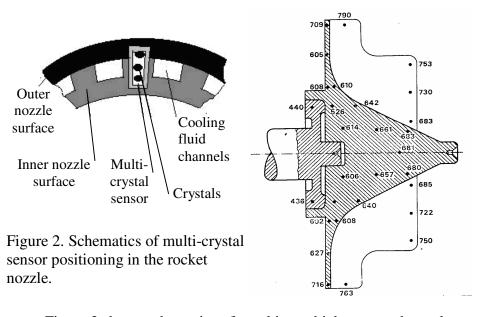


Figure 3. Rocket engine combustion chamber turbo pump cross-section showing sensor locations and corresponding measured temperatures in °C.

Figure 2 shows schematics of a turbine, which powers the rocket engine combustion chamber pump. Each of the 27 points in Figure 2 represents the temperature measured in °C. Sensors were placed on the opposite sides of the turbine diameter, in the center rod, disk and turbine blades, in order to get a better picture of temperature distribution [2].

In addition to the above tests, multiple measurements of space apparatus thermal protection systems were performed. The most interesting results were obtained from "BOR-4" and "BOR-5" spacecrafts, which were used for the flight tests of "BURAN's" thermal protection system [4, 5]. These measurements were performed in real conditions, on the orbit, as well as

during sub-orbital flights, descent and entry into atmosphere. "BOR-4" usually circled the Earth once, and "BOR-5" was launched on the sub-orbital trajectory with consequent entry into atmosphere followed by aerodynamic heating and parachute descent. Several launches were performed, and each spacecraft was equipped with 80 to 130 temperature sensors. The equivalent time was calculated to be on the order of 1 to 5 minutes for this non-stationary heating regime. Sensors were embedded at different depth into ceramic tiles, measuring temperatures from 150 to 1050 °C.

Another interesting sensor application was found in single crystal growth in space. Zero gravity allows growing better single crystals, as a problem of having heavier impurity atoms at the bottom of crystals grown on Earth is avoided. Crystal growth is greatly affected by the growth temperature, which needs to be carefully measured. As it is impractical to have heavy bulky temperature measurement systems in space, the lightest 3C-SiC temperature sensors were employed. Most of these measurements were conducted on the "MIR" space station [6]. Sensors were embedded along the furnace core, measuring the maximum and the minimum temperatures between 300 and 1000 °C with the heating time varying from 100 to 400 minutes.

# **CONCLUSIONS**

Irradiated single crystal sensors are the smallest maximum temperature sensors available. Due to their small size and weight they are very attractive for use in space applications. Several thousand 3C-SiC sensors have been used all over the world [7, 8]. Normally, "blind" tests were performed first, where several sensors were annealed in furnace with the time and qualitative thermal loading history being the only parameters given to researchers. After data reduction, the "true" temperature was revealed to the researchers. Table 2 shows such tests results, with standard deviation of  $\pm 5.6$  °C. Here, better results, compared to the specified sensor accuracy, given in Table 1, are explained by the fact that the tests were performed in stationary heating regimes.

Table 2. 3C-SiC sensors accuracy verification results.

Test temperature,	Number of	Temperature measured by	Country	Standard
°C	tested sensors	3C-SiC sensors, °C		Deviation, °C
179	3	183,185,190	USA	7
404	3	404,405,406	USA	1
406	3	408,410,408	Japan	2.7
579	3	579,580,577	USA	1
650	3	648,650,653	Sweden	1
698	4	699,697,698,694	Japan	1.5
737	3	739,742,736	USA	2.7
770	3	772,767,765	Sweden	3.3
866	5	877,877,873,870,877	Switzerland	8.8
870	3	876,869,875	Sweden	4
914	5	918,919,921,919,919	Switzerland	5.2
926	3	929,928,924	USA	2.3
987	3	987,987,983	Sweden	1.3
1070	5	1080,1084,1081,1077,1077	Switzerland	9.8
1079	5	1082,1082,1086,1082,1090	Switzerland	5.4

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