# Irradiated cubic single crystal SiC as a high temperature sensor

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## ABSTRACT

Radiation is known to cause point defects formation in different materials. In the case of cubic SiC single crystal radiation flux on the order of $2 \cdot 10^{20}$ neutrons/cm$^2$ at 0.18 MeV causes over 3% volume lattice expansion. Radiation-induced strain (measurable by X-Ray diffraction) can be relieved when the annealing temperature exceeds the temperature of irradiation. Based on this effect the original technology of maximum temperature measurement was developed a while ago. Single crystal SiC sensor small size (200-500 microns), wide temperature range (150-1450 °C), "no-lead" installation, and exceptional accuracy make it very attractive for use in small, rotating and "hard-to-access" parts, including, but not limited to gas turbine blades, space shuttle ceramic tiles, automobile engines, etc. With the advances in X-Ray diffraction measurements, crystal and thin film growth techniques, it is the time to revise and update this technology. Modeling radiation damage, as well as annealing effects are also beneficial.

## INTRODUCTION

Present paper is written to inform on merits and characteristics of a unique temperature sensor technology, and discuss plans for the related research and development efforts. Experimental verification of the Analytical Heat-Transfer Models is essential in modern engineering. Accurate knowledge of machine part temperature distributions enables the search for the best compromise between the contradictory requirements of machine's reliability, service life, and efficiency. A number of temperature measuring techniques are in use today in the industry and their advantages and limitations are well known to experts. These techniques include thermocouples (Slip-Ring Assembly or Radio Telemetry System), Optical Pyrometers, Thermo Paints [1] and Thermo Plugs [2]. SiC Single Crystal Max Temperature Sensor (Figure 1) is a new addition to this list with a distinctively different principle of operation and technical characteristics.

## SENSOR CHARACTERISTICS

Presented technology is based on the phenomenon of SiC crystal lattice expansion as a result of irradiation [3] and consequently relieving the acquired strain when the temperature of annealing exceeds the irradiation temperature [4]. It was developed in Russia 35 years ago [4-6], and is now well recognized in Europe and in the US as a viable alternative to the state-of-the art in temperature measurement for its cost effectiveness, miniature size, wide temperature range, high accuracy (±10 °C), and "no lead" installation. These sensors are most effective when used on small, rotating and "hard-to-access" parts, and are about 900 times smaller than the smallest Temp.-Plugs (Figure 1).

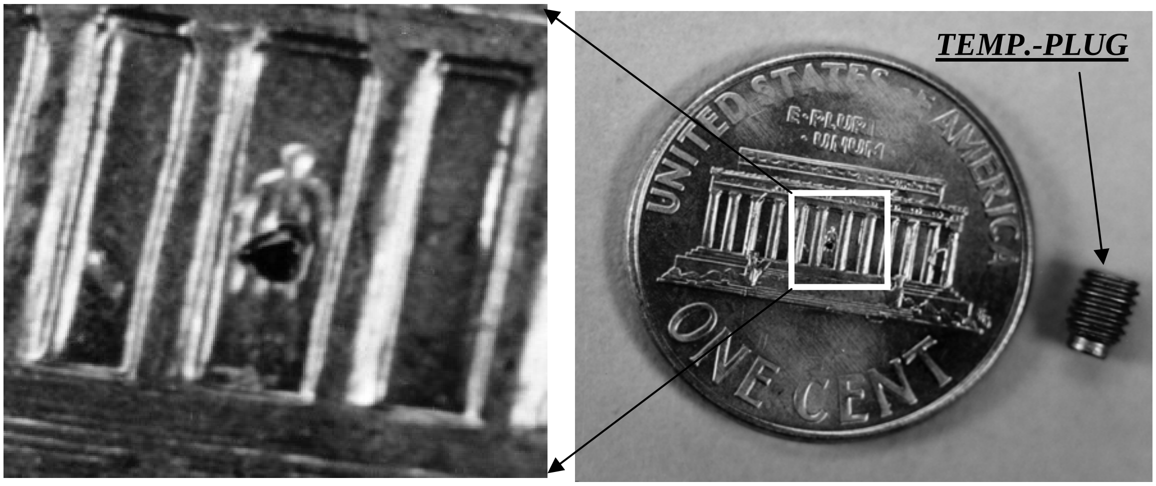

Figure 1. SiC crystal maximum temperature sensor along with a Temp.-Plug.

Maximum Temperature Crystal Sensor presents an elegant solution to a challenging technical problem. Accurate temperature measurements by miniature leadless sensors is possible due to the effective application of scientific principles of crystallography, nuclear physics, X-ray diffractometry, and heat-transfer.

## 3C-SiC irradiation induced defects and thermal annealing

Neutron irradiation interacts with SiC primarily by scattering off nuclei, an event, which suddenly imparts energy and momentum to an atom. If enough energy is transferred to exceed the displacement energy threshold, this scattering event creates radiation-induced defects. Defects are conveniently categorized into three types: point, line, and areal. In SiC, the important point defects are vacancies and interstitials; the line defects are threading dislocations; and the areal defects are stacking faults. One of the most important manifestations of lattice defects formation in SiC is the change in lattice volume. Theoretical and experimental investigations of 3C-SiC neutron irradiation process [5] have shown that up to 4% volume change, point defects clearly dominate the spectrum. One vacancy and one interstitial form a Frenkel Pair, which becomes a dominant defect formation mechanism if the number of created vacancies and interstitials are equal. Vacancy formation will decrease the lattice volume, while interstitials will increase it. Due to the fact that the absolute value of interstitial contribution is greater, formation of Frenkel Pairs will be followed by the resulting increase in lattice volume. Schematics in Figure 2 demonstrate the logic of irradiation parameters selection. Here, at 100 °C irradiation at $2*10^{20}$ neutrons/$cm^2$ flux and E>0.18 MeV, the resulting volume expansion is 3%-4%. Further increase in volume expansion will not improve sensor characteristics due to fact that beyond this point, the probability of complex defects formation becomes high. As a result of simultaneous presence of different types of defects in a crystal, two things will happen:

- relationship between lattice volume expansion and neutron flux will lose monotonous character, which could lead to results misinterpretation,
- root-mean-square value of atomic shift will grow too fast, adversely effecting intensity of Bragg reflection, which, in turn, will complicate lattice parameter measurements.

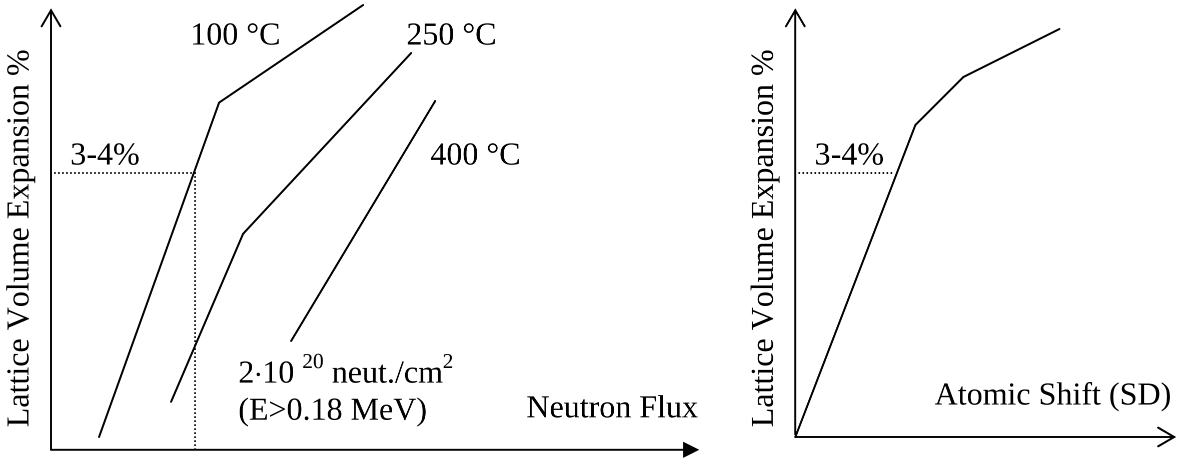

Figure 2. Schematic of lattice volume expansion as a function of neutron flux and irradiation temperature, and atomic shift.

Irradiation-induced defects in 3C-SiC crystals can be partially annihilated in a process of thermal annealing, which starts as soon as temperature of environment will exceed the temperature of irradiation (100 °C in this particular case). Annihilation of defects will cause lattice volume shrinkage, opposite to that caused by irradiation. Resulting change in lattice volume (measured with precision by means of X-ray diffraction) is a function of temperature and time of annealing. Fortunately, the time of annealing is easily recorded during the test, which makes the task of finding the maximum temperature simple and reliable.

Modern advancements in theoretical crystallography, nuclear science, and computer modeling [7] should be applied to simulate both irradiation and annealing processes. This will allow effectively optimize a choice of critical elements of crystal sensor manufacturing and application processes, (see Figure 3) such as:

- preferred SiC poly-type (3C vs. 6H or 4H) or even different compounds;
- the best way to introduce defects (neutron bombardment vs. gamma-rays, etc.);
- characteristics of defect formation procedure;
- calibration process parameters.

## Principles of sensor operation

Calibration of irradiated particles is performed by means of controlled annealing in lab conditions at different temperatures and times. The experimental information obtained in this way is used to form isochronal annealing curves (Figure 4a), which, in turn, will become the basis for creating the calibration nomogram similar to Figure 4b.

Later sensors are installed into machine parts using thermal cement, and, if necessary covered with nichrome foil. Sensor, embedded in a machine part, is exposed to the test environment. While the part is being tested, SiC crystal lattice undergoes transformation, which depends on the temperature and the duration of the test. X-ray diffractometer registers changes in lattice parameter compared to the initial unirradiated state, providing the information, which, together with the time history of annealing, allows determining the maximum temperature of the tested part. An x-ray beam of known wavelength is focused on a sample and a diffraction (reflection) angle is measured by a detector system. The d-spacing of the reflecting plane is calculated using Bragg's Law.

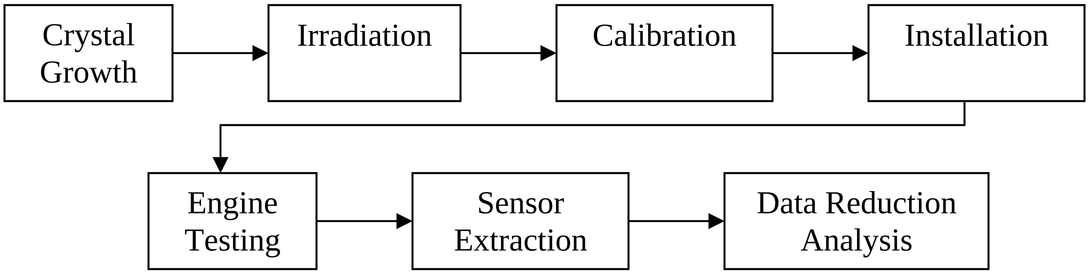

Figure 3. Block diagram of sensor manufacturing and application process.

Typical parts instrumented by crystal sensors are turbine/compressor blades, vanes, disks, shafts, seals, bearings, combustor liners, fuel injectors, afterburner components, exhaust nozzles, as well as reciprocating engine pistons, piston rings, combustor walls, spacecraft heat shields, and rocket nose cones.

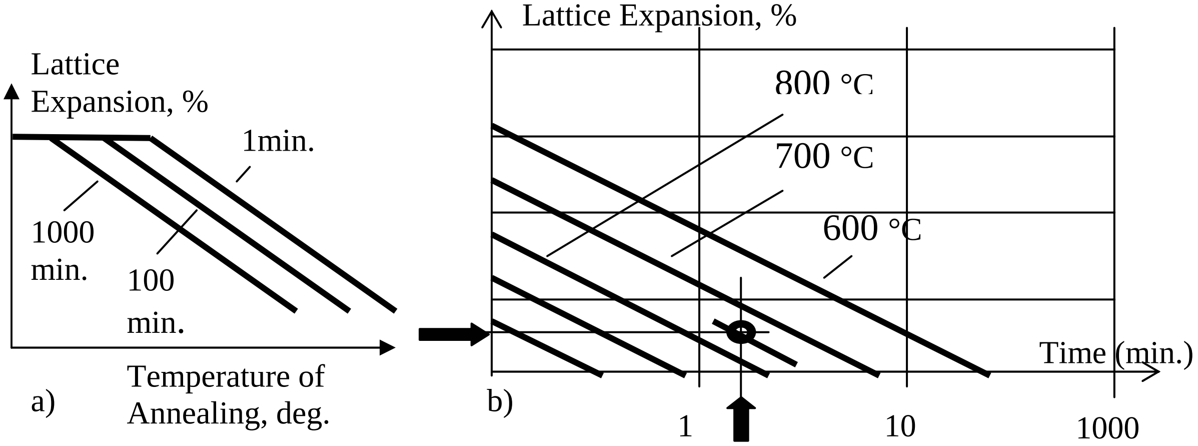

Figure 4. a) Curves of isochronal annealing; b) Calibration nomogram for the SiC temperature sensor.

## Measurement accuracy and comparison with competing techniques

Accuracy of this method had been thoroughly analyzed and compared with the characteristics of the competing technologies. According to [6], expected accuracy of the method (standard deviation, σ, in °C) can be assessed by using the following equation:

$$\sigma = \pm(-0.6 + 0.0071 \times T) \qquad (1),$$

where T is the maximum measured temperature in °C.

As demonstrated in Figure 5a, both Temp.-Plug and Thermal Paint methods are less accurate, then the SiC sensor. The most viable competitor is the Thermal Paint, where a temperature sensitive coating used to measure peak surface temperatures irreversibly changes of its color. It certainly will benefit those who need to obtain visual record of the temperature variations over the surface of components (parts), but numerous technological constrains, subjectivity in data interpretation, survivability issues, and tendency, as for any coating, to modify thermal behavior of a component during testing, will make it difficult to get accurate data. In addition, many so called "blind tests" had been performed recently by the companies using SiC sensor technology in Europe, Japan, and US. The term "Blind Test" refers to the fact that the SiC senor data reduction had been performed without any knowledge of the parallel measurement results obtained by well established techniques in close proximity to the sensor location. Comparisons had been made between the SiC sensor and Temp-Plugs on rotating disks, SiC sensor and stationary thermocouples on laboratory samples in an oven, SiC sensor and rotating thermocouples using slip-ring and telemetry. Similar studies were also done for a variety of temperature profiles, starting from the "ideal" long hold at $T_{max}$, to highly cyclical test configuration, shown in Figure 5b. Here, high temperature variations from 0.05 to 1 $T_{test}/T_{max}$ are plotted as a function of time. In all cases high accuracy was achieved, well in the bound of equation 1.

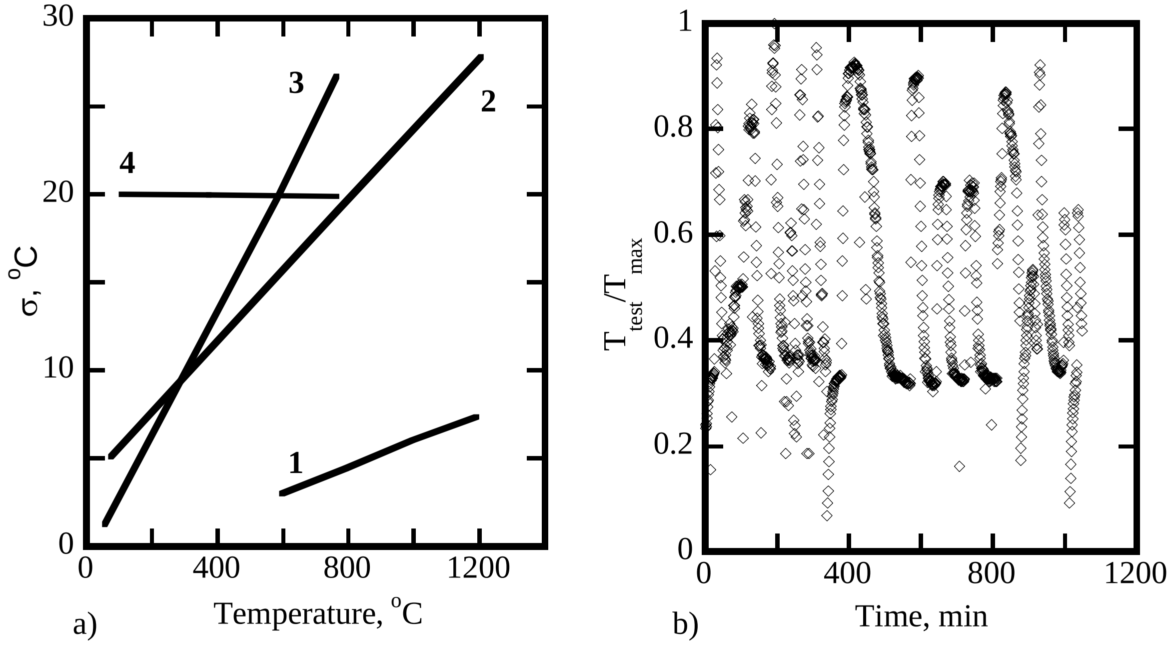

Figure 5. a) Temperature measurement accuracy for: 1-SiC sensor, 2-Thermal Paint (Thermocolor, Germany), 3-Thermal Paint (Riga), 4-Temp.-Plugs; b) Complicated temperature test profile.

## CONCLUSIONS

The idea of temperature measurement by means of irradiated crystals is proven and well accepted by the international scientific and engineering community. Technological upgrade will make the SiC single crystal temperature measurement technology even more attractive to traditional users. Further progress in micro-XRD and handling technologies should provide opportunities of entrance into the world of micro-electromechanical systems (MEMS) and nanoscale applications [8].

## REFERENCES

1. “Thermal Paint Technical Information”, Rolls-Royce Inc. (www.rolls-roice.com)
2. “Templug User Information Guide”, Rev.2.0, 1997, Testing-Engineers, Inc.
3. H. Huang, N. Ghoniem, J. of Nuclear Mater. 250, pp. 192-199 (1997)
4. V.A.Nikolaenko, V.A.Morozov, N.I.Kasianov “A Crystal Maximum Temperature Measurer for Special Applications.”-Rev. Int. Htes Temp. et Refract.1976, vol.13, pp.17-20
5. V.A. Nikolaenko, V.I. Karpuhin, “Measuring Temperature with Irradiated Materials”, “Izmerenie Temperaturi s Pomoschiu Obluchennih Materialov” Energoatomizdat, Moscow (1986)
6. V.N.Kuznetsov, V.A.Nikolaenko “Metrologia Metoda Izmerenia Maksimalnoi Temperaturi s Pomoschiu IMTK”, IAE-3208, Moscow (1979)
7. W.J. Weber, F. Gao, R. Devanathan, W. Jiang, Y. Zhang, “Experimental and Computational Studies of Ion-Solid Interactions in Silicon Carbide”, Mater. Res. Symp. Proc., Vol. 792, R5.1 (2004)
8. A.A. Volinsky, L.G. Ginzbursky, V.A. Morozov, “Crystal Temperature Sensor Technology Status and Future Research”, 2003 ASME Mechanics and Materials Conference book of abstracts, June 2003, Scottsdale AZ, 10 (2003)