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Influence of individual thermal shock parameters on stress generated in silicon nitride and its prediction

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Abstract

Influence of repeated thermal shock on the stress generated in silicon nitride, was determined by a new testing method. This method allows verification of temperature and stress progress obtained from a computer simulation. Input parameters were temperature, temperature difference, heating and cooling time. Output parameters were the mean stress and stress peaks of specific cycles. Two methods were used to compare the influence—a newly defined parameter of influence (PI) and a least square method. The results show a dominant influence of the temperature values, which is higher than the influence of temperature difference. The least squares method was also used to predict the value of stress, with coefficient of determination higher than 0.95.

Keywords: Thermal shock resistance; Testing; Si_3N_4

1. Introduction

Silicon nitride is known for its outstanding properties such as hardness, abrasion resistance, chemical stability, creep resistance, high-temperature strength (up to 1400 °C) and its resistance to thermal shocks. There are several methods used to test the resistance of silicon nitride to thermal shocks, among which the indentation-quench method is one of the most widely used for technical ceramics with high resistance to thermal shocks—since 1996[1] to the present time.[2,3] A number of modifications of this method have been introduced to lower the time consumption especially when dealing with repeated thermal shocks.[4–6] In the Department of Materials and Technologies of the Faculty of Mechanical Engineering of the Slovak University of Technology, a new testing method has been developed and optimized[7] together with a computer simulation of the temperature and stress conditions in the tested material.[8] This simulation makes it possible to evaluate the influence of thermal shock parameters on the stress, generated in the specimen. These parameters include the temperature difference, the absolute values of heating and cooling temperature and the heating and cooling time. The aim of our research is also the prediction of stress values, based on statistical analysis.

2. Experimental

Silicon nitride specimens were prepared by cold pressing and then hot pressing in nitrogen atmosphere. Activating densification aids for silicon nitride were Al_2O_3 and Y_2O_3 with a mass ratio corresponding to 10% of YAG. The hot pressing of the experimental material was performed on a laboratory hot press with a special construction for the heating body[9] (Fig. 1). This graphite body consists of two sections (1) and their electrical inputs (2) end with semicircular segments (3). These segments provide shielding of main electrical inputs (4) against heat radiated from the body.

The prepared specimens were of 2 mm thickness with 8 mm diameter. In the middle of the specimens, cracks were initiated using a Vicker's indentor. These cracks were used to determine the fracture toughness and were also important for the testing method itself. A similar method is being used also for indents, initiated using a hemispherical indentor.[10]

The depth profile of cracks was determined by horizontal serial sectioning of the material, as well as from the observation

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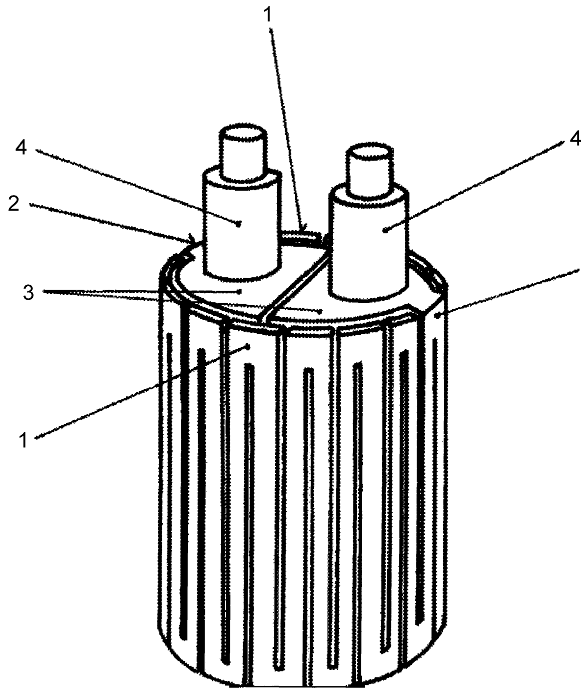

Fig. 1. Graphite heating body of the laboratory hot press. (1) Sections of the body, (2) electrical input to the heating body, (3) semicircular segments and (4) main electrical input.

of fracture areas. The sectioning took place in direction parallel with the surface of the specimen,[11] contrary to a previously used method,[12] where the direction of sectioning was perpendicular to the surface. Low speed of grinding discs, as well as rich lubrication ensure that the crack size does not increase during the sectioning. The depth profile is needed to specify the critical point for crack growth, which is located on the crack, under the deformed zone (Fig. 2).

The principle of the new testing method (protected by a patent[13]) is shown in Fig. 3. Specimens of circular cross-section (1) are being used in this test, with cracks (3) formerly initiated by Vicker's indentor. The specimen is placed with its damaged side (2) facing downwards and this side is constantly (without interruptions) cooled by water. Its opposite side is cyclically heated using a molybdenum punch (4). The punch is heated by an induction coil and is loaded by a mass of 6 kg. Its weight prevents the cooling water from leaking from under the sealing. The

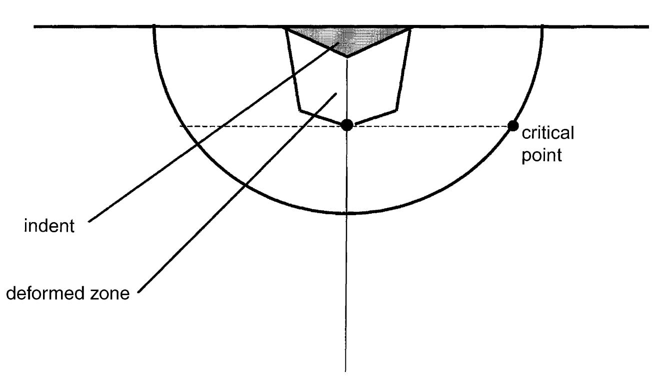

Fig. 2. Depth profile of the crack.

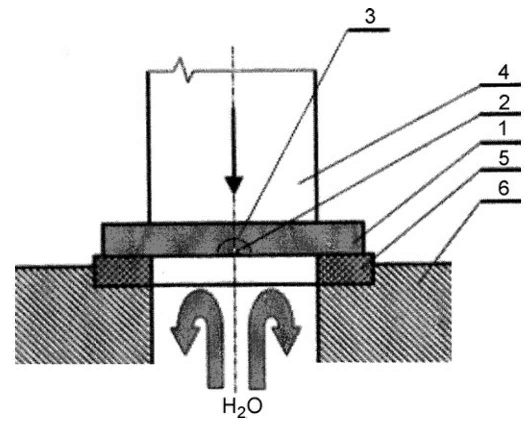

Fig. 3. Principle of the new method of testing the resistance of silicon nitride to repeated thermal shocks. (1) Specimen, (2) indent, (3) crack, (4) punch, (5) sealing, and (6) support.

diameter of the punch (5 mm) is equal to the hole diameter of the sealing. This way, the only mechanical loading of the specimen is shear acting under the perimeter of the punch. Mechanical loading acting on the indent and the cracks is negligible. The type of mechanical loading can be changed by modifying the punch diameter. The method is described more in detail in Refs. [14,15]. Test chamber of the device is made of steel, the transparent front wall, which allows observation, is made of temperature resistant glass. Rear wall, through which leads the induction coil, is made of thermally insulating material based on aramide fibers. Because the punch is made of molybdenum, the chamber must be evacuated before the test. During testing, protective gas (argon) is passing through the test chamber. Simulation and verification of temperature and stress progress has been introduced in Ref. [15].

The new testing method allows modification of various input parameters of the test. The reference parameters are as follows: heating temperature $T_h = 1100\,°C$, cooling temperature $T_c = 500\,°C$, heating time $t_h = 16\,s$ and cooling time $t_c = 6\,s$. These values provide the most intense thermal shock that can be achieved using our available equipment. For simplicity, a new method of description of input parameters has been introduced. Using this method, the reference parameters would be described as 1100/500-16/6.

The stress progress for these parameters for a specimen with thickness of 20 mm (in the critical point[15]—Fig. 2) is shown in Fig. 4. It can be seen, that the amplitude even in the first cycle is low, hence we can use the mean stress as representative for the entire stress progress. Thus, the first step was the evaluation of the influence of input parameters on the mean stress in a 20 mm thick specimen.

In Ref. [15], specimens with thickness of 2 mm were subject of a more thorough research and verification. The stress progress for this specimen thickness is shown in Fig. 5. It can be seen that even after 200 cycles the stress is not stabilized. This fact makes the evaluation of parameter influence more complicated. To facilitate the reproducibility of the results, it is necessary to study not only the mean stress, but also the value of stress in concrete cycles. This case is more common when dealing with

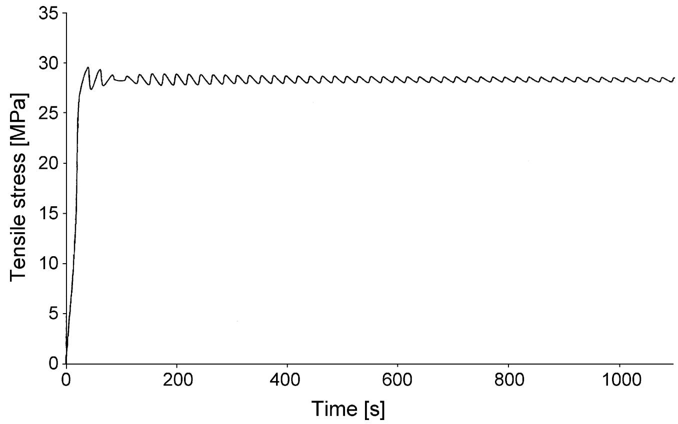

Fig. 4. Stress progress in critical point of a 20 mm thick specimen.

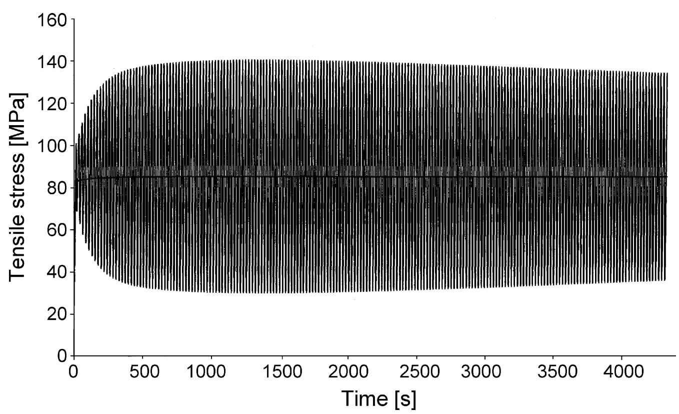

Fig. 5. Stress progress in critical point of a 2 mm thick specimen.

practical experiments, because mean stress is not responsible for unstable crack growth. We evaluated the influence of input parameters on the stress peaks in the 4th and 20th cycle.

For both specimen thicknesses, the influence of input parameters on the stress can be described by a so-called parameter of influence (PI). The PI is a ratio of the change of stress to the change of input parameter. This change (expressed in %) is understood with respect to the reference parameters. Thus, when looking for example for the influence of heating temperature on the value of stress, the PI will be calculated as:

$$\mathrm{PI} = \left| \frac{\Delta\sigma[\%]}{\Delta T_\mathrm{h}[\%]} \right| \quad (1)$$

Necessary condition when evaluating PI is that only one input parameter can be changed at a time. Other parameters must remain constant. The parameter of influence is only suitable for comparing of parameters' influence, it cannot be used to determine the absolute influence of a parameter. The evaluation using PI has been developed because we could not find a suitable widely used method. There was a possibility of using the ANOVA method[16] (analysis of variance), but this method only evaluates, whether or not a parameter has influence on the output quantity. It does not compare the influence of the parameters. Another disadvantage is, that it can only be used for cases with a finite number of values.

Our aim was not only to analyze the results, but also to predict the value of stress in the critical point. Based on the analysis of our results, most suitable method was the least squares method. Using this method, we were able to obtain the parameters' estimates in a linear regression model in the form:

$$\sigma = aT_\mathrm{h} + bT_\mathrm{c} + ct_\mathrm{h} + dt_\mathrm{c} + e + \varepsilon \quad (2)$$

where a, b, c, d and e are regression coefficients and ε is the normally distributed random error. This method is one of the methods commonly used for statistical analysis of results.[17]

3. Results and discussion

Results for 20 mm thick specimen are shown in Table 1. First line, containing reference parameters, is marked bold. Changed parameter is introduced in the third column, with its percentual change given in the fourth one. Most intense shocks, which can be experimentally achieved, are caused by reference parameters. For this reason the changed input parameters caused lower intensity of thermal shock. The influence of heating temperature was studied by decreasing T_h by 100 °C, maintaining constant temperature difference, hence the cooling temperature had to be lowered of the same amount as well (simulations 2 and 3). The influence of temperature difference was evaluated by increasing the cooling temperature while keeping the heating temperature constant (simulations 4 and 5). The influence of heating and cooling time was studied by increasing both quantities, lowering thus again the intensity of thermal shock (simulations 6–9). The next two columns of the table contain the values of the mean stress in critical point and the percentual change with respect to the value obtained at reference parameters. Parameter of influence (PI) is given in the last column. From Table 1 it can be

Table 1
Calculation of PI from simulations output for a 20 mm thick specimen

Simulation number	Input parameters	Changed parameter	Change of the parameter (%)	σ_mean	Change of σ_mean (%)	PI
1	**1100/500-16/6**	–	–	**28.97**	–	
2	1000/400-16/6	$T_\mathrm{h} = 1000$ °C	9.1	25.43	13.92	1.530
3	900/300-16/6	$T_\mathrm{h} = 900$ °C	18.2	21.87	32.46	1.784
4	1100/600-16/6	$\Delta T = 500$ °C	16.7	30.76	6.18	0.370
5	1100/700-16/6	$\Delta T = 400$ °C	33.3	32.53	12.29	0.369
6	1100/500-20/6	$t_\mathrm{h} = 20$ s	25	29.00	0.1	0.004
7	1100/500-25/6	$t_\mathrm{h} = 25$ s	56.3	28.98	0.03	0.001
8	1100/500-16/8	$t_\mathrm{c} = 8$ s	33.3	29.01	0.14	0.004
9	1100/500-16/10	$t_\mathrm{c} = 10$ s	66.7	29.00	0.1	0.002

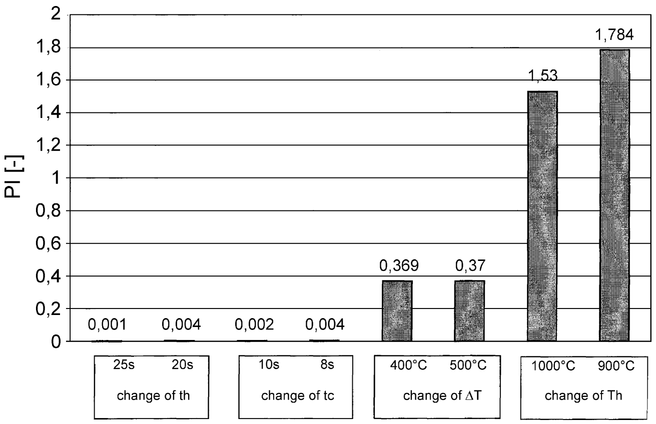

Fig. 6. Graphical representation of PI for a 20 mm thick specimen, the two rightmost columns showing the influence of change of heating temperature T_h.

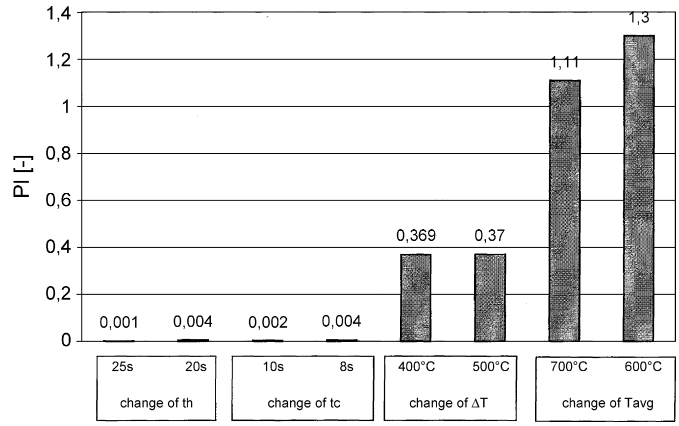

Fig. 7. Graphical representation of PI for a 20 mm thick specimen, the two rightmost columns showing the influence of change of average temperature T_{avg}.

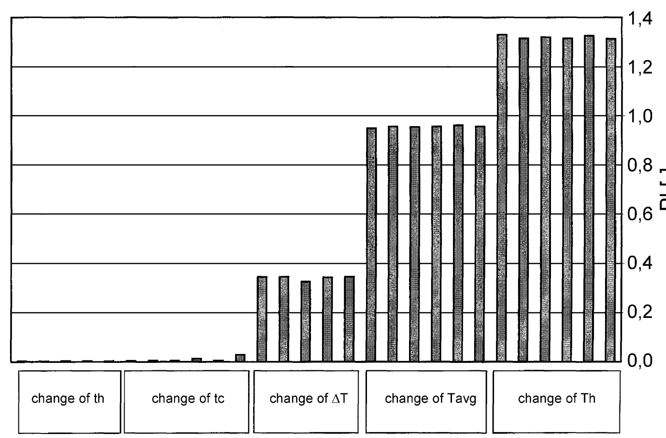

Fig. 8. Graphical representation of PI on mean stress for a 2 mm thick specimen.

seen that the reference parameters, which we stated as the most intense ones, caused a lower value of mean stress in most simulations (4–9). However, the reference parameters were determined as most intense not for mean stress, but for stress peaks.

Graphical representation of the results is shown in Fig. 6. It can be seen that the heating and cooling time has generally the lowest influence on the mean stress, PI is low because a significant change of these parameters caused only a negligible change of σ_{mean}. Most important conclusion from this series of simulations is that the temperature difference does not have the most significant influence on the resulting stress. PI reached its highest value with the change of heating temperature. The difference between the two values (1.53 and 1.784) represents 16.6%, which indicates only a relative suitability of PI.

The main reason for such a high PI in the case of change of heating temperature is probably that in fact two parameters (heating and cooling temperature) were changed at a time in order to maintain a constant temperature difference (Table 1, lines 2 and 3). However, during the definition of PI we declared that only one parameter can be changed at a time. A parameter which describes the change of both temperatures is in this case simply their average value T_{avg}. Parameters T_{max}, T_{min} and T_{avg} are characteristic for thermal fatigue. The aim of our work, however, is analysis of parameters within a small number of repeated thermal shocks, which cannot be considered as thermal fatigue, although our testing method is suitable also for this case. Let us replace lines 2 and 3 of the previous table.

Simulation number	Input parameters	Changed parameter	Change of the parameter (%)	σ_{mean}	Change of σ_{mean} (%)	PI
1	**1100/500-16/6**	$\mathbf{T_{avg} = 800\,°C}$	–	**28.97**	–	
2	1000/400-16/6	$T_{avg} = 700\,°C$	12.5	25.43	13.92	1.11
3	900/300-16/6	$T_{avg} = 600\,°C$	25	21.87	32.46	1.30

New results are shown in Fig. 7. The influence of temperatures, expressed by their average value, is lower. However, also the average temperature has more significant influence on the mean stress than the temperature difference. The difference between the values of PI (1.11 and 1.3) again casts doubt upon the PI method as an absolute of parameters' influence.

Next series of simulations was performed for a specimen with thickness of 2 mm. Because of a more complicated stress progress in this case, more simulations were performed for this thickness. The results for mean stress can be seen in Table 2 and Fig. 8. The change of both heating and cooling temperature (simulations 2–7) is again expressed also with the average temperature T_{avg} as an input parameter. The results for stress peaks in the 4th and 20th cycle are shown in Figs. 9 and 10.

The results show only small difference between the influence of parameters on the mean stress and on stress peaks in the 4th and 20th cycle. The only exception is the cooling time, especially when comparing its influence on the mean stress and on the stress in fourth cycle (Figs. 8 and 9). However, the influence is in both cases low, hence, this difference cannot be considered decisive.

The dominant influence of temperatures has been confirmed also when taking into account the average temperature as an input parameter. This can be considered a significant addition to

Table 2
Calculation of PI from simulations output for a 2 mm thick specimen

Simulation number	T_h (°C)	Changed parameter	Change of the parameter (%)	σ_{mean}	Change of σ_{mean} (%)	PI
1	**1100/500-16/6**	**–**		**95.625**		
2	800/200-16/6	$T_h = 800$ °C	27.3	61.319	35.88	1.314
		$T_{avg} = 500$ °C	37.5			0.957
3	850/250-16/6	$T_h = 850$ °C	22.7	66.847	30.09	1.326
		$T_{avg} = 550$ °C	31.3			0.961
4	900/300-16/6	$T_h = 900$ °C	18.2	72.752	23.92	1.314
		$T_{avg} = 600$ °C	25			0.957
5	950/350-16/6	$T_h = 950$ °C	13.6	78.470	17.94	1.319
		$T_{avg} = 650$ °C	18.8			0.954
6	1000/400-16/6	$T_h = 1000$ °C	9.1	84.188	11.96	1.314
		$T_{avg} = 700$ °C	12.5			0.957
7	1050/450-16/6	$T_h = 1050$ °C	4.5	89.906	5.98	1.329
		$T_{avg} = 750$ °C	6.3			0.949
8	1100/550-16/6	$\Delta T = 550$ °C	8.3	98.375	2.88	0.346
9	1100/600-16/6	$\Delta T = 500$ °C	16.7	101.126	5.75	0.344
10	1100/650-16/6	$\Delta T = 450$ °C	25	103.424	8.16	0.326
11	1100/700-16/6	$\Delta T = 400$ °C	33.3	106.627	11.51	0.346
12	1100/750-16/6	$\Delta T = 350$ °C	41.7	109.378	14.38	0.345
13	1100/500-16/7	$t_c = 7$ s	16.7	95.166	0.48	0.029
14	1100/500-16/8	$t_c = 8$ s	33.3	95.487	0.14	0.004
15	1100/500-16/9	$t_c = 9$ s	50	95.038	0.61	0.012
16	1100/500-16/10	$t_c = 10$ s	66.7	95.378	0.26	0.004
17	1100/500-16/11	$t_c = 11$ s	83.3	95.331	0.31	0.004
18	1100/500-16/12	$t_c = 12$ s	100	95.288	0.35	0.004
19	1100/500-18/6	$t_h = 18$ s	12.5	95.666	0.04	0.003
20	1100/500-20/6	$t_h = 20$ s	25	95.701	0.08	0.003
21	1100/500-22/6	$t_h = 22$ s	37.5	95.731	0.11	0.003
22	1100/500-24/6	$t_h = 24$ s	50	95.756	0.14	0.003
23	1100/500-25/6	$t_h = 25$ s	56.3	95.768	0.15	0.003

the "approach" used so far. Since The temperature difference has been considered not only as dominant, but as the only parameter of thermal shock when evaluating the resistance of technical ceramics to thermal shocks.[2,6,18–20]

The possibility to determine the influence of parameters on the stress becomes very complicated in the case when the mean stress in unstable (Fig. 11), because it is difficult to find a relevant value of the stress. The stress progress shown in Fig. 11 is from a point located on the axis of the specimen, 0.6 mm under the heated side of the specimen.

The number of simulations performed allows a statistical analysis of the results. Using least squares method, we obtained the parameters' estimates in the linear regression model for the mean stress in critical point and the resulting equations is:

$$\sigma_{mean} = 0.059T_h + 0.055T_c + 0.031t_h - 0.054t_c + 2.866 \quad (3)$$

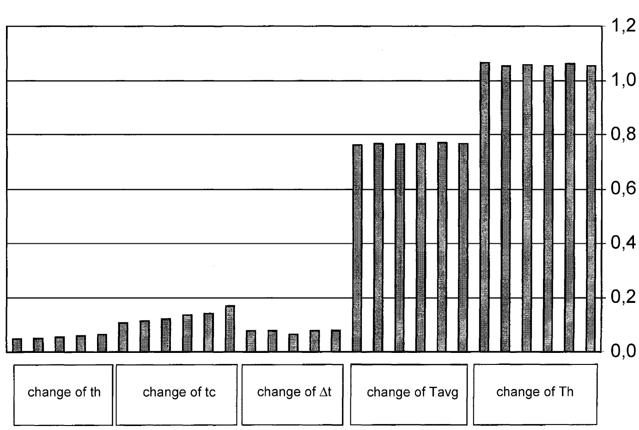

Fig. 9. Graphical representation of PI on stress peaks in the fourth cycle for a 2 mm thick specimen.

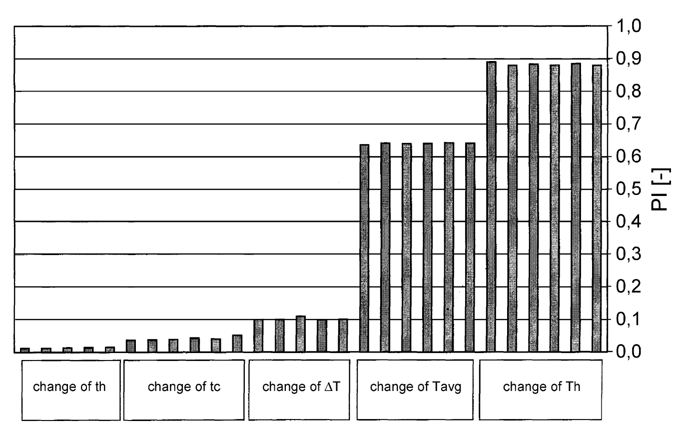

Fig. 10. Graphical representation of PI on stress peaks in the 20th cycle for a 2 mm thick specimen.

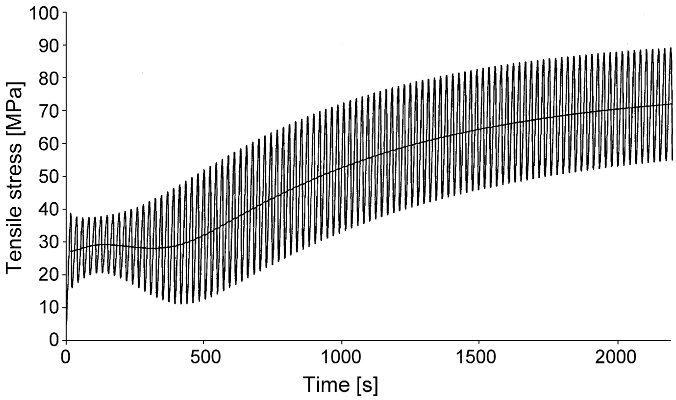

Fig. 11. Stress progress in a point located on the axis of 2 mm thick specimen, 0.6 mm under its heated side.

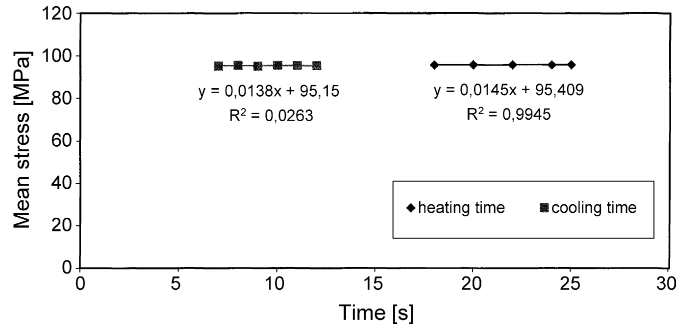

Fig. 13. Linearity of time influence on mean stress.

The results show a very good linearity—coefficient of determination for this model was 0.999855721, hence the equation can be considered reliable without having to "filter out" any errors using for example robust models.[21] Graphical representation of Eq. (3) would require a multidimensional graph. We chose a different approach to demonstrate the linearity of the parameters influence: a plot of stress versus one input parameter, while keeping the others constant. The linearity of influence of average temperature (at $\Delta T = \text{const}$) and cooling temperature (at $T_h = \text{const}$) is shown in Fig. 12 (for mean stress), along with the equations of their linear trend models and their coefficients of determination. The equations in this figure and the next ones contain variables x and y instead of the real physical quantities. This is a formal decision to prevent units mismatch. The slope of these lines also demonstrates the influence of the parameters. Again we must realize that the case of $T_h = \text{const}$ represents in fact the influence of ΔT. It proves true again that the influence of ΔT is lower than the influence of the absolute values of heating and cooling temperature. There is an unexpected influence of increasing cooling temperature (at $T_h = \text{const}$) on the mean stress. Increasing of cooling temperatures causes lower temperature difference, which should lead to lower values of mean stress.

The linearity of influence of heating and cooling time is shown in Fig. 13 and again demonstrates a negligible influence of these input parameters on the mean stress. Low value of the R-squared value of cooling time is caused by dispersion of the results, which corresponds also to the dispersion of PI for this case (Fig. 8 and Table 2, rows 13–18). In absolute values, however, this dispersion is negligible, as the maximum difference between the results is 0.449 MPa.

High correlation is maintained also when dealing with stress in a specific cycle. The resulting equations for the 4th and 20th cycle are stated below along with their coefficients of determination. Despite lower coefficient of determination in these cycles, its value is high enough to prove the equations reliable:

$$\sigma_{\text{4th cycle}} = 0.422T_h + 0.017T_c - 0.418t_h - 2.683t_c + 25.686,$$
$$\text{coefficient of determination} = 0.997279754 \quad (4)$$

$$\sigma_{\text{20th cycle}} = 2.288T_h + 1.9T_c - 1.158t_h - 9.798t_c + 104.59,$$
$$\text{coefficient of determination} = 0.965192316 \quad (5)$$

The linearity of input parameters influence in the fourth cycle is shown if Figs. 14 and 15. In this case there is an evident increase of the influence of cooling time, which corresponds to the results of PI in this cycle (Fig. 9). The slope of the cooling time's trend line is negative, but this does not indicate a lower influence—the influence is expressed by deviation of the trend line from its horizontal position. The influence of cooling temperature (influence of ΔT) on stress peaks in the fourth cycle is lower than its influence on the mean stress. This corresponds to

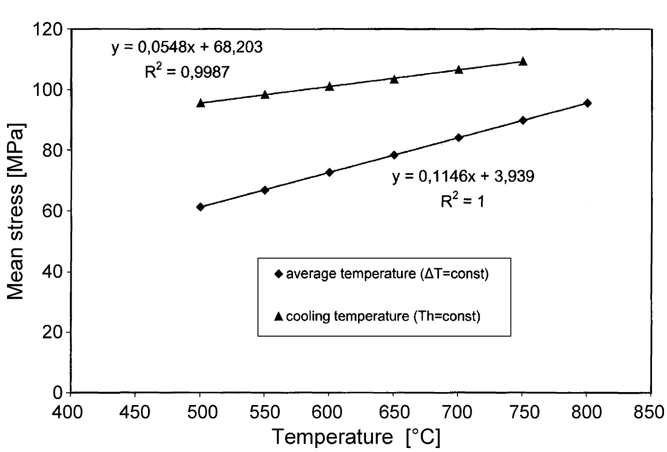

Fig. 12. Linearity of temperature influence on mean stress.

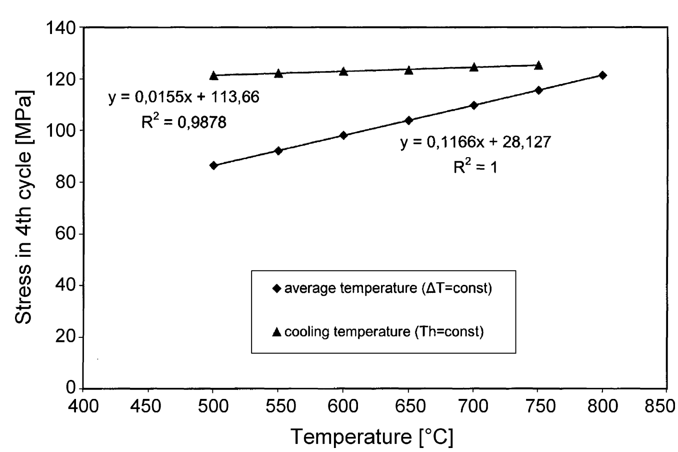

Fig. 14. Linearity of temperature influence on stress peaks in the fourth cycle.

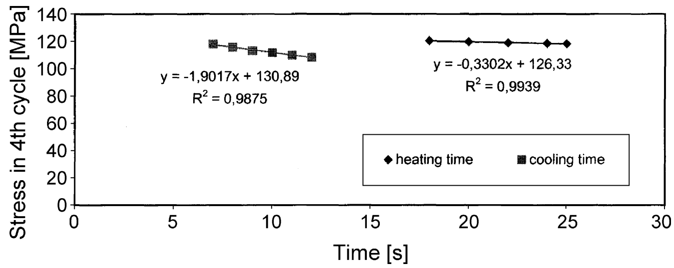

Fig. 15. Linearity of time influence on stress peaks in the fourth cycle.

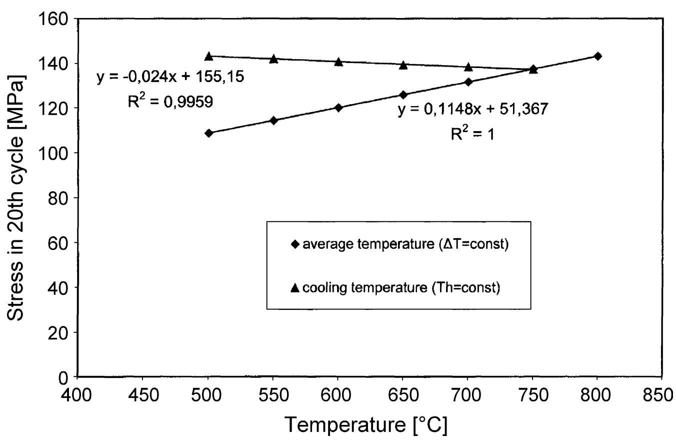

Fig. 16. Linearity of temperature influence on stress peaks in the 20th cycle.

the results of PI (Figs. 8 and 9). The coefficients of determination again exceed 0.95.

Figs. 16 and 17 demonstrate the linearity of input parameters influence in the 20th cycle. The influence of temperature is again dominant and the coefficients of determination are over 0.99. The main difference between the 4th and 20th cycle is the influence of the cooling temperature at $T_h = \text{const}$ (influence of ΔT). The influence of cooling temperature has an expected progress. Intersection of both lines in Fig. 16 is not of importance, it simply indicates that the stress peak in 20th cycle is for the simulations 1100/750 and 1050/450 almost the same (137.253 MPa and 137.464 MPa, respectively).

The influence of heating and cooling time on the stress peaks is in both 4th and 20th cycle negative, which is logical, because the intensity of thermal shock decreases with increasing time. From the point of view of reproducibility of the results, it is important to note the high values of reliability, represented by the coefficient of determination. Using the least squares method, we can easily obtain a model equation for any cycle and then estimate the value of stress in this particular cycle also for input parameters that have not been simulated.

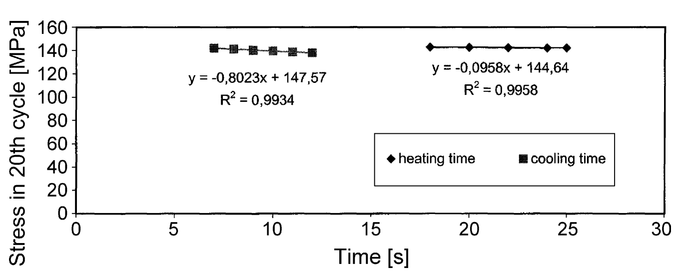

Fig. 17. Linearity of time influence on stress peaks in the 20th cycle.

The aim of this paper is an analysis of thermal shock parameters' influence in a point, which is critical for crack growth under the conditions of above described loading. Stress analysis was performed also for other 10 points located on vertical axis of the specimen, from the heated side down to the cooled side. Presentation of these results, however, would increase the volume of this paper too significantly and is beyond the scope of the presented topic.

The results of our analysis are generally applicable for any number of loading cycles. In the case of a specimen with 2 mm width, the value of stress peaks in the critical point becomes stabilized after 16 cycles,[15] hence thermal loading with number of cycles higher than this can be already considered as thermal fatigue. Based on general characteristics and terminology of thermal fatigue it is possible to define maximum stress, minimum stress and the mean stress.

4. Conclusion

The influence of repeated thermal shock parameters on stress, generated in silicon nitride was evaluated using parameter of influence (PI) and statistical model. The results show clearly a dominant influence of temperature, followed by the influence of temperature difference, and heating and cooling time. This result was the same for evaluation of the influence on mean stress and on the stress peaks in specific cycles.

The statistical model, obtained using least squares method, showed very high coefficient of determination. The highest value was reached by the equation for mean stress. Despite lower coefficients of equation for stress peaks in the 4th and 20th cycle, its value is high enough to prove the prediction reliable. The model confirmed the surprisingly lower influence of temperature difference on the stress and a very low influence of heating and cooling time.

It is hence important, at least in the conditions of loading described in this article, to take into account not only the temperature difference, but primarily the absolute values of heating and cooling temperature when evaluating the resistance of technical ceramics to repeated thermal shocks. The heating and cooling time should also not be neglected.

Presented results are applicable also for conditions of thermal fatigue.

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