



CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Ceramics International 37 (2011) 1985-1992

Development of input output relationships for self-healing Al₂O₃/SiC ceramic composites with Y₂O₃ additive using design of experiments

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 Received 29 September 2010; received in revised form 21 October 2010; accepted 13 February 2011
 Available online 8 April 2011

Abstract

 Al_2O_3/SiC ceramic composites with Y_2O_3 as an additive, was synthesized using the Taguchi method of design of experiments, so as to develop statistically sound input output relationships. The proportion of SiC was varied from 12 to 21 vol.% whereas that of Y_2O_3 was varied from 2.5 to 4 vol.%. The composites were sintered at 1500 °C for a soaking time period of 12 h in an air atmosphere. Cracks were induced on the composite surface using a Vickers indenter with a load varying between 20 and 40 kg. Fractographical analyses have been carried out using optical and/or scanning electron microscopy to investigate the surface crack propagation behavior. Thermal aging at 1300 °C in the time range of 0.5–12.5 h was applied to find optimal conditions for healing of the pre-cracked samples. The output parameters such as crack length, healed crack length, hardness and fracture toughness of the samples were correlated with appropriate inputs such as contents of SiC and Y_2O_3 , crack-healing temperature, healing time, compaction pressure, indentation load using statistical analysis. Further, the extent of influence, exerted by pertinent input parameters on output parameters, was also identified.

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Keywords: B. Composites; D. Al₂O₃; Crack healing; Fracture toughness

1. Introduction

In the class of engineering ceramics, alumina (Al_2O_3) is one of the most cost effective and widely used materials. Al_2O_3 due to its strong ionic bond provides desirable material properties. Nevertheless, it has some limitations too for being used as structural ceramic. However, it has three weak points viz. low bending strength (\sim 400 MPa), low fracture toughness (\sim 3 MPa m^{1/2}), and low heat-resistance limit (\sim 900 °C) for bending strength. These weaknesses restrict the application of Al_2O_3 as engineering ceramics [1,2]. In general, there are three ways to overcome the restriction [3–5], which are (a) non-destructive inspection [6] with an ability to repair the cracks, (b) enhanced fracture toughness of the ceramic matrix by reinforcing it with fibres/ whiskers, ductile or brittle particles, by effecting phase

transformation, crack tip shielding and crack deflection within the matrix [7] and (c) inducing a crack-healing ability to heal all the surface defects [8–11]. Many studies have been conducted on the techniques (a) and (b) mentioned above and useful results have been reported [12,13]. However, the technique (c) has only been studied by a few researchers. Applying the high crack-healing ability to ceramics and to structural components for engineering use could result in great benefits, such as an increased reliability, reduced inspection, machining and polishing costs of ceramic components. However, when the healing ability is considered in structural engineering, attention needs to be paid on many associated factors including the effect of chemical composition on the crack-healing ability, the effect of healing conditions on the strength of the healed zone, the maximum crack size that can be completely healed, knowledge of the high-temperature strength of crack-healed zones, the assessment of the cyclic-fatigue and static-fatigue strengths of a crack-healed ceramic component and crack-healing behavior during service [1]. From this point-of-view, the development and production of ceramic materials with a

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crack-healing ability is desired. Current ceramic matrix composite systems (CMCs) with a high crack-healing ability are mostly based on an oxide ceramic matrix with well-distributed SiC [7].

It is assumed that the crack healing is due to oxidation. The estimated crack-healing reaction is the following:

$$SiC + 2O_2 \rightarrow SiO_2 + CO_2$$
 (CO)

where, the oxygen that intrudes the cracked surface reacts with silicon carbide (SiC) present there. The extra bonding force exerted by the glass phase on the crack, either by transfer of the existing glass phase to the crack plane to bridge it [14] or owing to the reaction of the crack surface with the oxidation environment [15] bridges it. It appears that each of the above mechanisms takes place under specific conditions, i.e. heat treatment atmosphere and the presence of sintering additives, such as Y_2O_3 [16].

Whereas, a substantial literature on Al_2O_3/SiC composites having a crack-healing ability are available [5,7,17–19], studies on Al_2O_3/SiO_2 ceramic composites with Y_2O_3 additive having crack-healing ability are scanty [20,21].

Well-planned and designed experiments are powerful over the traditional approach of varying one-factor-at-a-time (OFAT) for experimentation. OFAT is unreliable, not costeffective and may lead to false optimal conditions. The design of experiment (DOE) is a structured, organized method that is used to determine the relationship between the different factors (Xs) affecting a process and the output (Ys) of that process. DOE involves designing a set of experiments, in which all relevant factors are varied systematically [22]. The analysis of results of these experiments helps to identify optimal conditions, the factors that most influence the results, and those that do not, as well as the existence of interactions and synergies between factors. DOE methods require wellstructured data matrices. Analysis of variance (ANOVA) delivers accurate results, when applied to a well-structured matrix, even when the matrix is quite small. The Taguchi method of experimental design (TMED) [23,24] is a typical fractional factorial design, which develops orthogonal arrays and produces same combination of experimental trials. These orthogonal arrays force all experimenters to design almost identical experiments. TMED seems to be the natural selection, since it is quite easy to understand. TMED produces a special set of orthogonal arrays and hence can also be called a Taguchi orthogonal array design (TOAD). Taguchi orthogonal arrays (OAs) are practical, simple and have been widely reported [25,26]. It is also important to achieve the objective of the experiment even having minimum budget and resources.

Sixteen types of Al₂O₃-based ceramic samples were prepared, by combining with SiC and Y2O3 (as a sintering additive) using the Taguchi method of design of experiments, so as to develop statistically sound input output relationships. The SiC content added was between 12 and 21 vol.% whereas Y₂O₃ was in the range of 2.5–4 vol.%. It is appropriate to mention that, to the best of the authors' knowledge, such studies on ceramic composites prepared in the present investigation have not been reported so far. The output parameters such as developed crack length, healed crack length, hardness, fracture toughness and density were correlated statistically with appropriate input parameters such as vol.% of both SiC and Y₂O₃, crack-healing temperature, healing time, compaction load, indentation load, etc. The order of significance of input parameters with respect to output parameters was also determined through the statistical analysis.

2. Design of experiments

In the present case, the ten steps of Taguchi's parameter design methodology [16] were used. Based on the input parameters shown in Table 1, the design table, shown in Table 2, has been developed. Based on the composition suggested in Table 2, sixteen different ceramic compositions were prepared, sintered and polished. The polished samples were indented and cracks were induced. The healing times for the cracks were taken from Table 2. Other fixed parameters for preparation of the samples are given in Table 3.

3. Materials and experimental method

Commercially available Al_2O_3 (99.8% purity, 0.4 μ m mean particle size, Industrial Ceramics) and SiC (95% purity, 63 μ m (220 mesh) mean particle size, Qualikems) were used as starting materials. Moreover, Y_2O_3 (99.99% purity, 0.3–.4 μ m, Himedia) was used as a sintering additive. Sixteen different compositions were prepared from the above three powders.

The required amount of the different powders, as given in Table 2, was mixed thoroughly in a liquid medium (acetone) for 12 h. The mixture was exposed to air for solvent evaporation resulting in a dry powder mixture. To this dry mixture, poly vinyl alcohol (PVA) binder was added (3% by weight). The mixture was subsequently compacted using a cold-uniaxial press, in a die of 16 mm diameter. The green compacted pellets

Table 1 Input and output parameters involved in ceramic composite design.

Input parameters (Xs)			Levels of factors			Output parameters (Ys)		
Name(s)	Туре	Unit	1	2	3	4	Name(s)	Unit
X_1 : SiC	Numeric	Vol.%	12	15	18	21	Crack length	μm
X_2 : Y_2O_3	Numeric	Vol.%	2.5	3.0	3.5	4.0	Healed crack length	μm
X_3 : compaction load	Numeric	Tonne	18.5	19.0	19.5	20.0	Density	g/cm ³
X_4 : healing time	Numeric	Н	0.5	4.5	8.5	12.5	·	_

Table 2 Taguchi's parameter design for the present study.

Run	SiC (vol.%)	Y ₂ O ₃ (vol.%)	Compaction load (Tonne)	Healing time (h)
1	12	2.5	18.5	0.5
2	12	3	19	4.5
3	12	3.5	19.5	8.5
4	12	4	20	12.5
5	15	2.5	19	8.5
6	15	3	18.5	12.5
7	15	3.5	20	0.5
8	15	4	19.5	4.5
9	18	2.5	19.5	12.5
10	18	3	20	8.5
11	18	3.5	18.5	4.5
12	18	4	19	0.5
13	21	2.5	20	4.5
14	21	3	19.5	0.5
15	21	3.5	19	12.5
16	21	4	18.5	8.5

were about 2.5 mm in thickness. Four pellets were made for each of the sixteen compositions. The pellets were sintered at $1500\,^{\circ}\text{C}$ for 12 h in an air atmosphere. Most of the SiC and Y_2O_3 particles were distributed uniformly in the grain boundaries.

Crack-healing conditions significantly affect the fracture behavior of crack-healed specimens. Thus the crack-healing behavior was systematically investigated as a function of crack-healing time (0.5–2.5 h) at a temperature of 1300 °C in an air atmosphere. The healing was a maximum at this temperature. Fig. 1c shows a specimen crack-healed at a temperature of 1300 °C, for a healing time of 0.5 h and in air environment.

3.1. Experimental method

The test specimens were surface ground and polished before testing. Cracks were introduced into the specimens by the indentation method using a Vickers indenter and applying different loads (20 kg, 30 kg and 40 kg). The shape of the cracks is shown in Fig. 1. The specimens were heat treated in air at 1300 °C for 0.5 h, 4.5 h, 8.5 h and 12.5 h to heal the crack. The heating rate was 10 K min $^{-1}$ and the samples were furnace cooled after the healing period.

Cracks were produced at the corners of the indentation impression. The crack length was found to vary with the

composition of the composites prepared, along with their load bearing capacity. The samples having compositions of 18 and 21 vol.% SiC were found to have lower fracture toughnesses, and were susceptible to failure at indentation loads (greater than 20 kg) and thus studies were limited to 20 kg. The radial crack emanating from the corners of the indentation impression can be confirmed from the specimen surface as shown in Fig. 1b.

4. Test results and discussion

Crack healing is sensitive to crack-healing conditions, such as temperature and time of the crack healing. SEM images of indentation with cracks, crack shape, filled up crack after the crack-healing treatment and glassy phase(s) [27] formed as a result of the heat treatment are shown in Fig. 1a-d respectively. A higher heating temperature and longer holding time promote the oxidation reaction of SiC. The oxidation results in silica either in amorphous or cristobalite forms, depending upon the temperature of operation. The amorphous silica, converts into a crystallized cristoballite form, above 1100 °C. Mullitization occurs due to the reaction between Al₂O₃ and the oxidationderived SiO₂ at 1400 °C. However, the presence of Y₂O₃ in Al₂O₃-SiO₂ system, lowers the mullitization temperature by about 100 °C. Mullitization is governed by a solution-precipitation control mechanism. The reaction between SiO₂ and Al₂O₃ produces a metastable eutectic liquid to form a transitory alumino silicate glass, which dissolves alumina giving rise to mullite precipitate. The eutectic compound (liquid) formed in the system Y₂O₃-SiO₂-Al₂O₃ has a low viscosity and therefore is able to flow into the pore channels thereby filling in some pores and cracks with this compound [1]. The evidence of the presence of glassy phase in a healed crack is also demonstrated by EDS analysis as depicted in Fig. 2a and b.

Table 4 shows the original and derived output parameters and their associated input parameters. Based on this table input–output correlations were developed using statistical analyses.

4.1. Development of correlation for prediction of average crack length

Taguchi's method of experiment design consisting of a set of 16 experiments as given in Table 2 were performed and the output parameters were noted. One of the output parameters of the present investigation is the average crack length and this is

Table 3 Other pertinent fixed parameters for the preparation of $Al_2O_3/SiC-Y_2O_3$ composite ceramics.

S. no.	Fixed parameters	Unit	Value/name
1.	Type of solvent	_	Acetone
2.	Amount of binder (poly vinyl alcohol)	wt.%	3
3.	Blending time	h	12 (magnetic stirring)
4.	Sintering temperature	$^{\circ}\mathrm{C}$	1500
5.	Sintering time (in air)	h	12
6.	Crack-healing temperature	$^{\circ}\mathrm{C}$	1300
7.	Size of sample in the shape of a cylindrical pellet	mm	16 mm diameter, 2.5 mm thickness

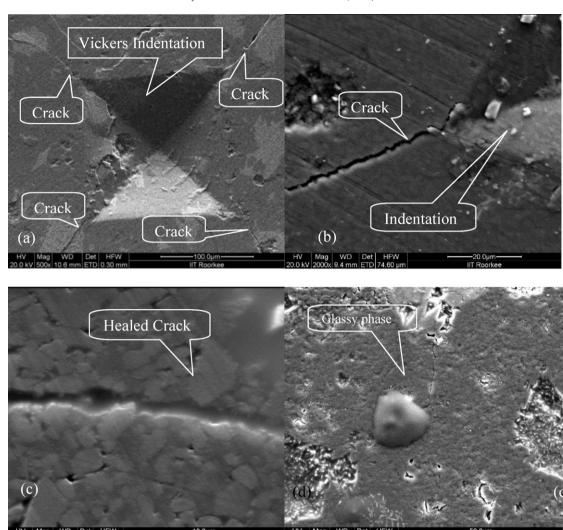


Fig. 1. SEM micrographs of the sample surfaces showing (a) indentation with cracks, (b) crack shape, (c) healed crack after heat treatment and (d) glassy phase formed after heat treatment.

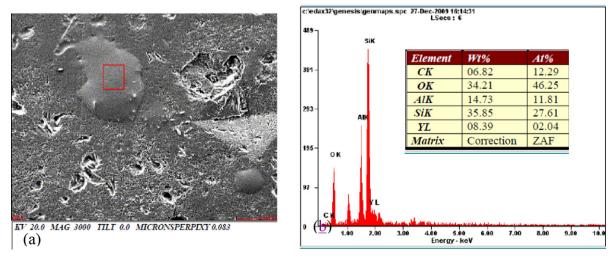


Fig. 2. (a) Micrograph of a healed crack surface showing glassy phase formed and (b) EDS analysis result of the glassy phase through area scan.

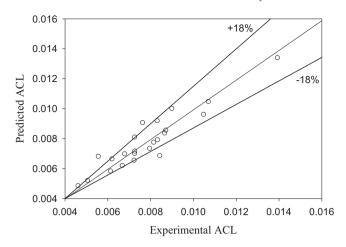


Fig. 3. Parity plot for the reduced quadratic model, Eq. (6), developed for prediction of yield (wt.%).

used for development of regression models. The different proposed models namely linear, 2-factor interaction and quadratic as shown in Table 5 are used for development of input-output correlations.

As all the input parameters used for the development of correlations should be independent of each other, therefore, before proceeding for the regression analysis the correlations between the input parameters in terms of a correlation coefficient were determined and are reported in Table 6. The value of the correlation coefficients ranges from -1 (a perfect inverse relationship) to +1 (a perfect direct relationship). A value of 0 indicates no relationship. The correlations coefficients in Table 6 show that no correlations exist between input parameters as coefficients are near to a zero value.

Statistical testing of the models were performed with an F-test to obtain the mathematical relationship between input and output parameters. An F-test is a statistical test in which the test statistics has an F-distribution under the null hypothesis. It is most often used for comparing statistical models that have been fitted to a data set, in order to identify the model that best fits the population from which the data were sampled. To examine the goodness of fit of a model, the test for significance of the regression model was performed and ANOVA is applied to the response data.

4.1.1. Case I: development of linear model

First of all, a linear model is fitted considering four input parameters and the regressed linear model is given by Eq. (2). An ANOVA was performed to establish the relative significance of the individual factors and is given in Table 7. In ANOVA, the p-value determines whether a model and model terms are significant (p < 0.05) or insignificant. The correlation coeffi-

Table 4 Original and derived output parameters.

S. no.	Output parameter(s)	Units	Input parameter(s)					
			SiC (vol.%)	Y ₂ O ₃ (vol.%)	Compaction load	Healing time	Indentation load	
			$\overline{X_1}$	X_2	X_3	X_4	<i>X</i> ₅	
1 2 3 4 5	Average crack length (ACL) Healed crack length (HCL) Vickers hardness (H_V) Fracture toughness (FT) Density	μm μm GPa MPa m ^{1/2} g/cm ³	\ \ \ \	\ \ \ \ \	\ \ \ \ \	√	√ √	

Table 5
Generic forms of models investigated in the present study.

S. no.	Name of the model	Generic form
1	Linear model	$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \dots + \beta_5 X_5$ where $Y = \text{response}$; β_0, \dots, β_5 are regression coefficients; X_1, \dots, X_5 are regression variables
2 3	2-factor interaction model Quadratic model	$Y = \text{linear model} + \beta_{12}X_1X_2 + \dots + \beta_{15}X_1X_5 + \beta_{23}X_2X_3 + \dots + \beta_{25}X_2X_5 + \dots + \beta_{45}X_4X_5$ $Y = 2 - \text{factor interaction model} + \beta_{11}X_1^2 + \beta_{22}X_2^2 + \dots + \beta_{55}X_5^2 \dots$

Table 6 Correlation analysis of the input parameters.

	X_1 : SiC	X_2 : Y_2O_3	X_3 : compaction load	X_5 : indentation load
X ₁ : SiC	1			
X_2 : Y_2O_3	0.059324	1		
X_3 : compaction load	0.074853	0.27005	1	
X_5 : load	-0.48867	-0.25941	-0.16744	1

Table 7 ANOVA of regression analysis for Eq. (4).

Predictor	Coefficient	Std. error	t	p	Significance
Constant	-3.6	191.1	-0.02	0.985	Insignificant
X_1 : SiC	9.952	2.170	4.59	0.000	Significant
X_2 : Y_2O_3	-7.300	7.723	-0.95	0.355	Insignificant
X_3 : compaction load	-2.275	9.769	-0.23	0.818	Insignificant
X_5 : indentation load	3.0315	0.9031	3.36	0.003	Significant
R-square: 55.3%	F-value (for model, Eq. (4)): 6.51 p-value (for model, Eq. (4): 0.001				

Table 8 ANOVA of regression analysis for Eq. (5).

Predictor	Coefficient	Std. error	t	p	Significance
Constant	-1012.7	569.2	-1.78	0.091	Insignificant
X_1 : SiC	-25.562	6.909	-3.70	0.002	Significant
X_2 : Y_2O_3	2.913	5.822	0.50	0.623	Insignificant
X ₃ : compaction Load	70.19	30.42	2.31	0.032	Significant
X_5 : load	48.16	23.09	2.09	0.051	Significant
$X_1 \times X_5$	1.6137	0.3066	5.26	0.000	Significant
$X_3 \times X_5$	-3.341	1.271	-2.63	0.017	Significant
R-square: 82.0%	F-value (for model 1	Eq. (5)): 14.39			-
•	<i>p</i> -value (for model,	Eq. (5)): 0.0001			

cients of the regression analysis along with other pertinent parameters are given in Table 7.

Average crack length

$$= -4 + 9.95X_1 - 7.30X_2 - 2.27X_3 + 3.03X_5 \dots \tag{4}$$

Now, from Table 7, it is clear that X_1 and X_5 are the significant parameters whereas, the rest are insignificant. From Table 7, it can be seen that Eq. (4) offers an around R-square (the coefficient of determination) value equal to 55.3% which is less and thus, the present model is not a good model. Thus there is a need to examine the next model.

4.1.2. Case II: development of a 2-factor interaction model

The 2-factor interaction (2FI) model is developed using the experimental average crack length (ACL) values. The regressed

2FI model was found to be inadequate in explaining the value of ACL as it offered predicted R-square (42.57%) far less than R-square (84.5%). Thus, the regression equation (Eq. (5)) for the reduced 2FI model was tried, by dropping the insignificant terms in a stepwise manner. An ANOVA was performed to establish the relative significance of the individual factors and is given in Table 8.

Average crack length

$$= -1013 - 25.6X_1 + 2.91X_2 + 70.2X_3 + 48.2X_5$$

+ 1.61X₁X₅ - 3.34X₃X₅... (5)

From Table 8 it can be seen that X_1 , X_3 , X_5 , X_1X_5 , X_3X_5 are significant whereas X_2 is insignificant. Further, the R-square value is 82% and p-value is also significant. Thus this appears to

Table 9 ANOVA of regression analysis for Eq. (6).

Predictor	Coefficient	Std. error	t	p	Significance
Constant	-309.47	4540	-0.0682	0.946	Insignificant
X_1 : SiC	-20.722	8.61	-2.4069	0.0294	Significant
X_2 : Y_2O_3	12.0974	4.69	2.5775	0.021	Significant
X_3 : compaction load	-16.5337	468.92	-0.0353	0.972	Insignificant
X_5 : indentation load	49.3504	23.84	2.30	0.0562	Significant
$X_1 imes X_5$	1.3810	0.415	3.33	0.0046	Significant
$X_3 \times X_5$	-3.22	1.366	-2.83	0.0324	Significant
X_3^2	-2.2965	12.078	0.1901	0.8518	Insignificant
R-square: 86.8%	<i>F</i> -value (for model <i>p</i> -value (for model,	•			

Table 10 Final input–output correlations for Al₂O₃/SiC plus Y₂O₃ ceramic composites.

S. no.	Output parameter	Correlation	$R_{\rm sq.}$, $F_{\rm value}$, $p_{\rm value}$
1.	Avg. crack length (ACL)	$1/ACL = 1/(-309.47 - 20.722X_1 + 12.0974X_2 - 16.5337X_3 + 49.3504X_5 + 1.3810X_1X_5 - 3.22X_3X_5 - 2.2965X_3^2)\%$ Error based on parity plot: $\pm 18\%$	$R_{\text{sq.}} = 86.8\% \ F_{\text{value}} = 14.10 \ p_{\text{value}} < 0.0001$
2.	Healed crack length (HCL)	$\begin{aligned} & \text{HCL} = -1719.3099 + 79.1485X_1 + 346.7439X_2 + 28.7262X_5 \\ & + 22.3797X_4 - 8.4345X_1X_2 - 4.5944X_2X_5 - 0.6442X_5X_4 \\ & - 1.3886X_4X_1 - 1.0659X_1^2 - 11.8152X_2^2 - 0.0888X_5^2 + 0.9731X_4^2\% \\ & \text{Error based on parity plot: } -15\% \text{ to } +20\% \end{aligned}$	$R_{\text{sq.}} = 90.9\% \ F_{\text{value}} = 4.99 \ p_{\text{value}} = 0.0297$
3.	Vickers hardness (H_V)	$H_{\rm V} = -1.3111 - 0.0003X_1 + 0.0033X_2 - 0.0008X_2^2 + 0.2027X_3 \\ -0.0105X_3^2 + 0.000014X_3X_1 + 0.000065X_2^3 + 0.0002X_3^3\% \\ {\rm Error\ based\ on\ parity\ plot:} \ \pm 10\%$	$R_{\text{sq.}} = 93.5\% \ F_{\text{value}} = 20.6 \ p_{\text{value}} < 0.0001$
4.	Fracture toughness (FT)	FT = $0.6504X_1 - 0.3561X_3 + 0.0169X_1^2 + 0.0727X_2^2 + 0.0264X_3^2 - 0.0448X_1X_2 - 0.0516X_3X_1\%$ Error based on parity plot: $\pm 20\%$	$R_{\text{sq.}} = 90.1\% \ F_{\text{value}} = 21.26 \ p_{\text{value}} < 0.0001$
5.	Density	Density = $128.4425 + 0.0239X_1 - 37.2432X_2 + 2.1609X_2^2 - 8.9731X_3 + 0.1295X_3^2 + 0.0007X_1X_2 + 1.9196X_2X_3 - 0.0058X_2^2X_3^2\%$ Error based on parity plot: $\pm 2\%$	$R_{\text{sq.}} = 95.8\% \ F_{\text{value}} = 14.32 \ p_{\text{value}} = 0.0047$

be a good model. However, the third alternate model should be checked for betterment.

4.1.3. Case III: development of quadratic model

The quadratic model is developed using the experimental average crack length values. The regressed quadratic model was found to be inadequate in explaining the value of ACL. Thus, the regression equation for the reduced quadratic model was tried by dropping the insignificant terms in a stepwise manner, and is given by Eq. (6). An ANOVA was performed to establish the relative significance of the individual factors and is given in Table 9.

$$1/ACL = 1/(-309.47 - 20.722X_1 + 12.0974X_2$$
$$-16.5337X_3 + 49.3504X_5 + 1.3810X_1X_5$$
$$-3.22X_3X_5 - 2.2965X_3^2) \dots$$
(6)

From Table 9, it is seen that X_1 , X_2 , X_1X_5 and X_3X_5 are significant and all other terms are insignificant. The R-square value, which is equal to 86.8% is better. The model p-value is also significant. Thus, this model can be used to navigate the design space. Therefore, taking all the facts into consideration, it can be concluded that the reduced quadratic model is the best model.

A parity plot for the reduced quadratic model given by Eq. (6) for the prediction of ACL was created and is shown in Fig. 3. The error band extends from -18% to +18%, and 91% data points fall within this error band. Thus, the average crack length predicted by Eq. (6), lie within -18% to +18% of experimental values.

Similarly, based on the above analysis methodology, regressed correlations were developed for output parameters such as healed crack length, Vickers hardness, fracture toughness and density in a stepwise manner. However, due the paucity of space the final correlations with other pertinent parameters are only shown below in Table 10.

Table 11
The order of influence of input parameters with respect to output parameters.

Output	Order of influence of input parameters					
parameter	1	2	3	4	5	
ACL	X_1X_5	X_1	X_3X_5	X_5	-	
HCL	X_4^2	X_1	X_5X_4	X_4X_1	X_2X_5	
$H_{ m V}$	X_3	X_3^2	X_3^3	X_3X_1	X_{2}^{3}	
FT	X_1^2	X_1X_2	X_2^2	X_1X_3	-	
Density	X_3	X_2	$X_2X_3^2$	X_2^2	$X_2^2 X_3^2$	

The order of influence of input parameters on different output parameters have been estimated from the regression analysis and are presented below in Table 11.

From Table 11 it is clear that for output ACL, HCL, H_v , FT and density, the most influential input parameters, which contribute a maximum, are vol.% of SiC × indentation load, healing time, compaction load, vol.% SiC and compaction load respectively. Further, for the above five output parameters in the order given above the second most influential input parameters are vol.% SiC, compaction load, vol.% SiC × vol.% Y_2O_3 and vol.% Y_2O_3 respectively. Similarly, the third, fourth and fifth most influential input parameters or their combinations for all the five output parameters are shown in Table 11. This information can be effectively used to control output parameters of the experiment.

5. Conclusions

Sixteen different samples of Al₂O₃–SiC–Y₂O₃ ceramic composites were prepared using the Taguchi method of design of experiments. The original output parameters such as average crack length, healed crack length, density and derived output parameters such as Vickers hardness, and fracture toughness were correlated successfully using regression analysis with input parameters such as vol.% of SiC, vol.%

of Y_2O_3 , compaction load and healing time. These correlations can be used for mathematical modeling of Al_2O_3 –SiC– Y_2O_3 ceramic composites to predict their properties if these are created under the same experimental conditions as the present one.

The first, second, third, fourth and fifth most influential input parameters or their combinations have been identified which can be used to control all the five output parameters such as ACL, HCL, H_v , FT and density of the experiment effectively.

References

- S.P. Liu, K. Ando, B.S. Kim, K. Takahashi, In situ crack-healing behavior of Al₂O₃/SiC composite ceramics under cyclic-fatigue strength, Int. Commun. Heat Mass Transfer 36 (2009) 558–562.
- [2] W. Nakao, M. Ono, S.K. Lee, K. Takahashi, K. Ando, Critical crack-healing condition for SiC whisker reinforced alumina under stress, J. Eur. Ceram. Soc. 25 (2005) 3649–3655.
- [3] K. Niihara, New design concept of structural ceramic nanocomposites, J. Ceram. Soc. Jpn. 99 (1991) 974–982.
- [4] J. Zhao, L.C. Steams, M.P. Harmer, H.M. Chan, G.A. Miller, Mechanical behavior of alumina–silicon carbide nanocomposites, J. Am. Ceram. Soc. 76 (1993) 503–510.
- [5] H.S. Kim, M.K. Kim, S.B. Kang, S.H. Ahn, K.W. Nam, Bending strength and crack-healing behavior of Al₂O₃/SiC composites ceramics, Mater. Sci. Eng. A 483–484 (2008) 672–675.
- [6] K. Ando, Y. Shirai, L.M. Nakatan, Y. Kobayashi, S. Sato, (Crack-healing + proof test): a new methodology to guarantee the structural integrity of a ceramics component, J. Eur. Ceram. Soc. 22 (2002) 121–128.
- [7] W. Nakao, K. Takahashi, K. Ando, Threshold stress during crack-healing treatment of structural ceramics having the crack-healing ability, Mater. Lett. 61 (2007) 2711–2713.
- [8] L. Hollaway, Polymer Composites for Civil and Structural Engineering, Chapman & Hall, London, 1993.
- [9] M.C. Chu, S. Sato, Y. Kobayashi, K. Ando, Damage healing and strengthening behavior in intelligent mullite/SiC ceramics, Fatigue Fract. Eng. Mater. Struct. 18–19 (1995) 1019–1029.
- [10] K. Ando, T. Ikeda, S. Sato, E. Yao, Y. Kobayashi, A preliminary study on crack healing behavior of Si₃N₄/SiC composite ceramics, Fatigue Fract. Eng. Mater. Struct. 21 (1998) 119–122.
- [11] K. Ando, K. Houjyou, M.C. Chu, S. Takeshita, K. Takahashi, S. Sakamoto, S. Sato, Crack-healing behavior of Si₃N₄/SiC ceramics under stress and

- fatigue strength at the temperature of healing (1000 $^{\circ}\text{C}$), J. Eur. Ceram. Soc. 22 (2002) 1339–1346.
- [12] G.C. Wei, P.F. Becher, Development of SiC-whisker-reinforced ceramics, Am. Ceram. Soc. Bull. 64 (1985) 298–304.
- [13] H.E. Kim, A.J. Moorhead, Oxidation behaviour and effects of oxidation on the strength of SiC-whisker reinforced alumina, J. Mater. Sci. 29 (1994) 1656–1661.
- [14] T.K. Gupta, Crack healing and strengthening of thermally shocked alumina, J. Am. Ceram. Soc. 59 (1976) 259–262.
- [15] H.W. Kim, H.E. Kim, H. Song, J. Ha, Effect of oxidation on the room-temperature flexural strength of reaction-bonded silicon carbides, J. Am. Ceram. Soc. 82 (1999) 1601–1604.
- [16] K. Houjou, K. Ando, M.C. Chu, S.P. Liu, S. Sato, Effect of sintering additives on the oxidation behavior of $\rm Si_3N_4$ ceramics at 1300 °C, J. Eur. Ceram. Soc. 25 (2005) 559–567.
- [17] N.L. Han, Z.G. Wang, L.Z. Sun, Effect of reinforcement size on low-cycle fatigue behavior of SiC particle-reinforced aluminum-matrix composites, Scripta Metall. Mater. 33 (1995) 781–787.
- [18] S.P. Liu, K. Ando, B.S. Kim, K. Takahashi, In situ crack-healing behavior of Al₂O₃/SiC composite ceramics under static fatigue strength, Int. Commun. Heat Mass Transfer 36 (2009) 563–568.
- [19] Z. Chlup, P. Flasar, A. Kotoji, I. Dlouhy, Fracture behaviour of Al₂O₃/SiC nanocomposite ceramics after crack healing treatment, J. Eur. Ceram. Soc. 28 (2008) 1073–1077.
- [20] T. Grande, H. Sommerset, E. Hagen, K. Wiik, M.A. Einarsrud, Effect of weight loss on liquid-phase sintered silicon carbide, J. Am. Ceram. Soc. 80 (1997) 1047–1052.
- [21] S. Baud, F. Thévenot, A. Pisch, C. Chatillon, High temperature sintering of SiC with oxide additives: I. Analysis in the SiC-Al₂O₃ and SiC-Al₂O₃-Y₂O₃ systems, J. Eur. Ceram. Soc. 23 (2003) 1–8.
- [22] D.C. Montgomery, Design and Analysis of experiments, 5th ed., Wiley-Interscience, 2004.
- [23] J. Antony, D. Perry, C. Wang, M. Kumar, An application of Taguchi method of experimental design for new product design and development process, Int. J. Assembly Technol. Manag. 26 (2006) 18–24.
- [24] G. Taguchi, S. Chowdhury, Y. Wu, Taguchi's Quality Engineering Handbook, John Wiley & Sons, NJ, 2005.
- [25] X. Chen, Y. Zhang, G. Pickrell, J. Antony, Experimental design in fiber optic sensor development, Int. J. Prod. Perform. Manag. 53 (2004) 713– 725
- [26] M. Jahanshahi, M.H. Sanati, S. Hajizadeh, Z. Babaei, Gelatin nanoparticle fabrication and optimization of the particle size, Phys. Status Solidi (a) 205 (2008) 2898–2902.
- [27] S. Ding, S. Zhu, Y. Zeng, D. Jiang, Effect of Y₂O₃ addition on the properties of reaction-bonded porous SiC ceramics, Ceram. Int. 32 (2006) 461–466.