REFRACTORY AND CERAMIC MATERIALS

CRACK RESISTANCE OF MODERN CERAMICS AND CERAMIC COMPOSITES. II. EF METHOD

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UDC 621.762

The procedure and test results are for crack resistance are presented for various monolithic and composite oxide and nonoxide ceramics by chipping the rectangular edge of a polished specimen. The main difference of the test method suggested (EF method) with an arbitrary point of fracture for a specimen edge from the well-known similar method with a fixed point of fracture is demonstrated. Tests are performed with Rockwell, Vickers and Knoop indenters, and the Rockwell indenter is chosen as the optimum. On the basis of statistically reliable experimental data a direct relationship is established between edge toughness and critical fracture toughness determined in the same ceramic specimens based on scandium oxide, aluminum oxide, zirconium dioxide, silicon carbide and silicon nitride. The EF method, whose use does not require special equipment, may be used effectively in a normal metallurgy test laboratory, particularly when specimens for evaluating ceramic breaking resistance may only be prepared in smaller sizes than those required for standard crack resistance tests.

Keywords: ceramics, fracture, chip, indenter, toughness, edge toughness.

INTRODUCTION

Ceramic materials are irreplaceable in many fields of technology, in medicine and in everyday life. They, as for the first ceramic, created in the ancient past and changing the way of human life, are brittle with a tendency towards uncontrollable breakage. This feature of the mechanical behavior of these materials limits their application, not only in highly stressed structures, but also in many other cases since an unexpected critical force upon them cannot be excluded. As ceramics have become more improved and have found more extensive application there has been an increase in attention to studying their breaking resistance and the development of appropriate test methods. As has been shown in [1], the SEVNB method may be considered the optimum method for evaluating the crack resistance of various ceramics, in accordance with which a specimen in the form of a beam with a V-shaped stress concentrator breaks in bending. However, even this method is not always applicable for studying functional, nanostructural and other similar materials. For example, this is mainly due to the expenditure of material in preparing specimens of a standard size that often appear to be larger than the article for which the material is intended.

The IF method does not solve the problem of evaluating crack resistance by indenting a polished surface with a Vickers indenter [1]. A feature of the IF-method is local material breakage and analysis of the stopped cracks. It has been shown in [3, 4] that the critical stress intensity factor determined by this method may differ considerably from the corresponding values that are obtained by a method based on studying failure of a specimen with a propagating crack. If attention is drawn to the difficulty of measuring the size of a crack formed at the corners of an indentation with tests on heterogeneous ceramics by the IF method, then it is possible to agree with the opinion in [5] about the usefulness of this method for comparative evaluation of the resistance of ceramics to breaking only within the same laboratory.

Institute of Strength Problems, National Academy of Sciences of Ukraine, Kiev. Translated from Poroshkovaya Metallurgiya, Nos. 7-8(450), pp. 39-49, July-August, 2006. Original article submitted February 16, 2005.

Fig. 1. Diagram of the method of specimen edge chipping (a) and a chip at the edge of a boron carbide specimen (b): d is distance from the point of loading to the specimen edge, P_f is loading force, D is chip height, W is maximum chip width, K is chip length

In contrast to this, the EF-method [6], according to which the indenter chips the rectangular edge of a polished specimen, combines the positive features of known test methods for crack resistance: a study of crack propagation in a specimen and breaking of a small volume of material. The present article, supplementing work in [1], is devoted to the EF method, probably similar to the method that was used to prepare weapons and tools in the Paleolithic period and not based on the ideas of linear fracture mechanics [7].

Interest in using the chipping effect in tests for crack resistance has been caused by the publication in [8] whose authors recalled similar work in the distant past and suggested use of a polycrystalline diamond indenter as a chipping tool applying a static load to a specimen. They also established constancy for the ratio of chip dimensions with a change in load causing breaking of a specimen edge. These authors developed [9] a method for determining resistance to breakage of brittle materials (from glass to metal carbides) with chipping of a specimen edge. As a characteristic of crack resistance they chose edge toughness M (N/mm) equal to the ratio of load P_f causing splitting of the edge of a rectangular specimen to the distance of its point of application to this edge (d in Fig. 1a). A connection was shown between values of edge toughness M and the rate of elastic energy liberation G_{Ic} [7].

Similar work has also been carried out in the National Physical Laboratory of Great Britain [10]. Here Rockwell and Knoop indenters were used. The results obtained confirmed the existence of the relationship $M - G_{Ic}$ for different brittle materials (including a brittle tool steel) and they indicate that no connection between the edge toughness M and the critical stress intensity factor K_{Ic} is observed.

The authors in [11] studied the edge toughness of sintered silicon nitride ceramic. The effect of the edge angle size for a chipped specimen on the results of determining M was established and on the basis of this a construction of a valve for an internal combustion engine made of this ceramic was established.

In studies in [12] of stomatological ceramic, by determining the edge toughness using a procedure similar to [9], a standard Rockwell indenter was replaced by a diamond cone. The recorded relationship $P_f - d$ did not appear to be linear, which was particularly prominent in analyzing test results corresponding to the low level of specimen loading.

Work in [13] is also well known whose authors introduced a Rockwell indenter into the polished surface of a specimen and moved it in the direction of a specimen edge up to the instant of chipping. Then the length of the chip K was measured (Fig. 1b) and the critical stress intensity factor was calculated by an equation close to that used in determining crack resistance by the IF method [14]. Values presented by the authors for critical stress intensity factor for four ceramic material, tested previously [15], correlated with values of obtained by the SEVNB method. However, we have not been able to reproduce these results using the same materials as the authors in [13], studied in accordance with the program in [15]. Therefore this work is not discussed further.

Proceeding from the aforementioned we note that although interesting results have been obtained in carrying out studies attention has not been devoted to the features of the mechanical behavior of ceramics [16], and in formulating

Fig. 2. Dependence of edge toughness M on the rate of energy release G_{Ic} for ceramics [9, 10]

Fig. 3. Specimen of GPSSN ceramic after testing by the EF method

Fig. 4. Diagram of the chip zone in the direction of indentation (a) and a chip in a silicon carbide specimen ST-TM (b)

common features of the class of brittle materials ceramics have not been separated. This is therefore possible if, for example on the basis of test data in [9, 10], an attempt is made to construct the dependence of chip toughness M on the rate of elastic energy liberation G_{Ic} only for ceramics excluding the rest of the brittle materials, then a field of points is obtained not relating each other (Fig. 2). This conclusion was made also by the authors in [13]. Therefore in [17] development of a new method for determining ceramic resistance to breaking was started and continued in [6], that in [18] was called the EF (edge fracture) method.

PROCEDURE AND MATERIALS

In determining the resistance of brittle materials to failure by the EF method at first close to the rectangular edge of a polished specimen a point was selected visually (or by means of a magnifier) for introducing the indenter to which load P_f was applied causing chipping of part of the edge. This was repeated several times (Fig. 3). Then by means of a binocular microscope Olympus BX51 the distance of breaking L was measured (Fig. 4a) considering it as a chipping characteristic. The point of the indenting was moved along the edge of the specimen (distance L was changed) randomly since visually it is impossible to orientate it accurately, and correspondingly changed the load P_f . The ratio of this load to the breaking distance L was considered as chipping toughness E_t . The value of E_t was determined by means of linear regression analysis [19]. The relationship $P_f - L$ (hereinafter the failure diagram) for the test material is constant (Fig. 5) and it makes it possible to consider the value of E_t its mechanical property. The EF method may be conditionally called a chipping method with an arbitrary loading point.

Fig. 5. Failure diagram for nanostructural ceramic 3Y-TZP (1) and polycrystalline silicon (2): $E_{IR} = 787$ (1) and 165 N/mm (2)

TABLE 1. Ceramic Properties

Ceramic	Index	Density, kg/m ³	Measure of brittleness χ	K_{Ic} , MPa · m ^{1/2}	Compaction technology	Publication
Silicon nitride	GPSSN	3.23	1	5.36	Sintering under pressure	[1, 6, 15]
	Si ₃ N ₄	3.16	1	5.5	Hot compaction	[17]
Zirconium dioxide	Y-PSZ	6.05	1	4.96	Sintering	[1, 6]
	Mg-PSZ	5.62	0.42		“	[1, 16]
Aluminum oxide	Al ₂ O ₃	3.70	1	2.93	“	-
Silicon carbide	SC-F	3.15	1	2.5	Sintering under pressure	[6]
Scandium oxide	Sc ₂ O ₃	3.79	1	1.49	Sintering	[20]

In order to carry out tests a universal unit Ceram Test [1] was used within which a two-coordinate table was mounted and Rockwell, Knoop, and Vickers indenters were installed. Specimens on the two-coordinate table were fastened by means of special grip. The rate of movement of the traverse of the test machine (and correspondingly the indenter) during a test was constant and it was 0.5 mm/min.

For more complete understanding of the features of the EF method for experiments markedly differing ceramics were selected (Table 1) prepared under laboratory and industrial conditions. Ceramics prepared under industrial conditions were aluminum oxide (Shanghai Morgan Macros Technical Ceramics Co. Ltd.), zirconium dioxide, partly stabilized with magnesium oxide (Nilcra Corp.), silicon carbide (ESK Ceramics GmbH and Co. KG). In the majority of cases the ceramics were isotropic and they deformed elastically up to breaking. In other words their mechanical behavior corresponded to the model of a solid adopted in linear failure mechanics [7]. We note that materials were selected similarly used in the Round Robins studies* connected with estimates of ceramic crack resistance [21].

Considering that critical stress intensity factors (Table 1) play an important role in analyzing the results obtained the value of them was determined by the SEVNB method [1], and in fragments of specimens, prepared here, the basic determination of chipping toughness was carried out in this work. We note that in preparing specimens their surface after grinding polished with diamond paste with an average grain size of 2 μ m, and the edge radius normally did not exceed 20 μ m.

RESULTS AND ANALYSIS

The main attention was devoted to studies within which a conical Rockwell indenter was used providing formation of a round impression since in this case no problems arise with the accuracy of orientation for specimens with respect to the diagonal (rib) which is typical for other indenters. At the same time for a complete picture of the possibilities of the EF method tests were performed within which for edge chipping of specimens different indenters

* Prestandard studies organized by the Japanese Society for Fine Ceramics (JECC), and also the international organizations VAMAS and ESIS.

were used applied in determining ceramic hardness and crack resistance. These studies (Fig. 6) made it possible to conclude that any indenter is suitable for these tests. We note that although in using different indenters the failure diagrams may be linear, there appeared to be a difference in the values of chipping toughness E_c . In order to exclude the possibility of confusion in test results it was suggested that appropriate indices were introduced into chipping toughness [6], for example in the case of using a Rockwell indenter this value was designated as E_{cR} .

In exploratory studies for the breaking resistance of ceramics it was detected that in some cases comparatively considerable scatter is observed for experimental data and therefore attention was devoted to this question. In studies of homogeneous silicon nitride ceramic (for example GPSSN in Fig. 7a) the scatter of experimental results was limited. However, in tests on heterogeneous ceramic SC-F it appeared to be much greater (Fig. 7b). Here the increase in stress concentration in the failure zone (increase in indenter sharpness) for this ceramic led to a reduction in the scatter of test data, that was not observed in testing ceramic GPSSN. We also note that this scatter is not connected with a scale effect (tests on specimens of ceramics SC-F and SC-TM with a cross section of $3 \times 3 \text{ mm}^2$ and $9 \times 10 \text{ mm}^2$, respectively). However, in the course of tests it became clear that the scatter of data observed by us was less than that published by other authors [10] for silicon carbide ceramic. This also points in favor of the EF method according to which attention is devoted to the actual chip size formed at a specimen edge and not an idealized picture created due to using the value of d .

We note that in tests with heterogeneous materials with uncontrolled installation of the Rockwell indenter in the grip of the loading unit scatter of test data varied somewhat on rotation with respect to the axis. This is possibly connected with the nonideal shape of the conical surface of the Rockwell indenter or presence within it of some defects that are not excluded for indenters of domestic production (the requirement for using high quality indenters was also drawn to attention in [9]). Therefore in the most critical tests a Rockwell GD indenter manufactured to special order by the American firm Gilmore Diamond Tools was used.

In the present work without upsetting the tradition established by the authors in [9], within whose tests it was only possible to operate with the surface of a specimen into which an indenter was introduced, as a failure parameter we adopted the breaking distance L (Fig. 4a) measured in this surface. Considering that in measuring the loading force on a Rockwell indenter proportionality should not be upset for the ratio of chip dimensions formed in a specimen [8], the corresponding study was carried out. It was established that instead of the value of L , actually equal to chip thickness, it

Fig. 6. Failure diagrams for tool silicon nitride ceramic obtained with specimen edge chipping by different indenters

Fig. 7. Failure diagrams for ceramics GPSSN (a) and SC-F (b) with different indenters: 1) $E_{tR} = 583$ N/mm, 2) $E_{tV2} = 590$, 3) $E_{tK} = 470$, 4) $E_{tV1} = 480$, 5) $E_{tR} = 417$, 6) $E_{tK} = 274$ N/mm

Fig. 8. Dependence of maximum chip width on breaking distance L (a) and also the failure load on distance L (width W) (b) for GPSSN ceramic

Fig. 9. Failure diagrams for hot-pressed ceramic Si_3N_4 and a laminar ceramic composite $Si_3N_4/30\% TiN + Si_3N_4$ in directions A and B

is possible to use its maximum width W (Fig. 1b) and to obtain a linear relationship $P_f - W$ (Fig. 8a) with breaking of a specimen edge by different indenters. It is important to note that on the basis of these experiments it is also possible to determine the value of chipping toughness (E_{tR}^* in Fig. 8b).

The EF method appeared to be useful in studying the breaking resistance of anisotropic materials. For example, it was revealed that for hot-pressed silicon nitride ceramic the value of chipping toughness is less in the compaction direction than in a direction perpendicular to it (Fig. 9a). No less interesting are the results obtained in studying a laminar ceramic composite $Si_3N_4/Si_3N_4 + 30\% TiN$ with rigid interlayer bonds within which the outer compressed layer was prepared from the above mentioned silicon nitride ceramic [22]. The actual breaking resistance determined by the EF method of this outer compressed layer was established to be reduced due to the action of internal tensile stresses (Fig. 9b). Therefore, the operating efficiency of these composites is not evaluated correctly on the basis of their effective

Fig. 10. Failure diagram (a) and R -curve (b) for Mg – PSZ ceramic [16]: Δl is specimen deflection in bending

Fig. 11. Dependence of chipping toughness E_{IR} on the critical stress intensity factor K_{IC} for different ceramics (statistically reliable data)

crack resistance determined by normal methods (results are provided in [1] for testing these composites by the SEVNB method), and there is no sense in preparing, for example, a cutting tool from them.

We note that all of the ceramics under discussion were linearly-elastic and with regression analysis the relationship $P_f - L$ was described by an equation for a straight line $P_f = a + E_t \cdot L$ (a is load with crossing of the coordinate axis for this value). In tests of rather brittle ceramic Mg – PSZ, phase changing in the stress field (Table 1), and ceramic $\text{La}_{0.8}\text{Ca}_{0.2}\text{CoO}_3$ [23], for which on loading there is typically rotation of ferroelastic domains (its measure of brittleness $\chi = 0.22$ [16]), these relationships did not appear to be linear (Fig. 10a). In this case the chipping toughness should in fact be determined on the tangent to the relationship $P_f - L$, and consequently it will increase with an increase in breaking distance L . In other words, an effect is observed (Fig. 10b) similar to the effect described by the R -curve [7]. If data provided in Fig. 10a are approximated by sections of a straight line, by separating the relationship $P_f - L$, for example in the ranges 0.15-0.3, 0.30-0.45, and 0.45-0.60 mm, then the value of E_{IR} will equal 550, 891, and 1040 N/mm, respectively. Therefore, the correctness of determining chipping toughness (the same as the critical stress intensity factor in standard and other tests) for these materials appears to be questionable. We note that in studying the failure resistance of ceramics by known methods the R -curve may only be established with particularly labor-intensive studies, and with use of the EF method it is easy to detect during analysis of the results of normal studies.

Proceeding from this study it is desirable to compare the results of determining chipping toughness (EF method) and the critical stress intensity factor (SEVNB method) in the same specimens (Fig. 11). By analyzing these statistically reliable data (average values of E_{IR} correspond to 50-100 or more experimental determinations) it is possible to conclude that the results of tests for material crack resistance carried out by different methods are comparable. Consequently, there is a basis for assuming that the EF method is suitable for comparative estimates of ceramic failure resistance.

CONCLUSIONS

Tests have been carried out on ceramic materials for crack resistance. It has been shown that the EF method may be used in determining the resistance of such materials to breakage.

In tests by the EF method a small volume of material is required and therefore its use, for example, is attractive in studying expensive nanostructural materials, and also in all cases when standard specimens used for these purposes are greater in size than the articles for which the materials being evaluated are intended.

The EF method is simple in application, no special equipment is required and therefore its use is possible in material science laboratories.

The specimens for study were kindly supplied by scientific colleagues in different countries: Jacob Kubler (Switzerland), Jannet Quinn (USA), Michael Swain (Australia), Andreus Rendtel (Germany) and others who the author thanks. The main experiments were performed in the Institute of Strength Problems, National Academy of Sciences of Ukraine, by aspirant Sergei Mudrik and also engineers Vasilii Galenko and Boris Ozerskii.

The work was partly financed by an INCO-Copernicus grant (Contract ICA2-CT-2000-10020).

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