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# Physical and Metallurgical Characteristics of Fiber Reinforced Ceramic Matrix Composites

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## 1. Introduction

Ceramics represent a very wide range of materials, which differ from each other by their chemical composition, structure and properties. These materials can be defined as predominantly crystalline materials, formed mainly by inorganic compounds of non-metallic character, which are bound particularly by ionic and ion-covalent bonds.

Ceramics are most frequently divided by the purpose of its use into **traditional** and **structural ceramics**. Traditional ceramics are produced entirely from natural raw materials (such as kaolin, clays, quartz, feldspar), which are formed and fired.

These ceramic materials can also be designated as vitreous or porous due to the fact that they contain a considerable portion of vitreous phase, pores and crystals of various compositions. Traditional ceramics can be further divided into constructional, artistic, utility and refractory.

Structural ceramics are produced only from synthetic raw materials. It is a newer type of ceramic, which has a considerable development from the seventies of the 20th century. It is used in numerous technical applications, especially under extreme conditions where, thanks to its properties, completely replaces the traditional ceramics. These ceramic materials include also functional and bio-ceramic materials.

Structural ceramics represent a fine-grained, poly-crystalline material, usually with grains smaller than 1  $\mu\text{m}$ . It contains random technological defects and micro-structural heterogeneities. The structure of ceramic materials can be characterized as **heterogeneous, poly-crystalline and poly-phased**.

## 2. Classification of structural ceramics

### 2.1 Oxidic ceramics

The ceramics based on aluminium oxide ( $\text{Al}_2\text{O}_3$ ) called also sintered alumina ( $\text{Al}_2\text{O}_3$  with addition of  $\text{ZrO}_2$ ) has the biggest significance for structural applications. Thanks to the zirconium oxide this ceramics has better mechanical properties. Increasing the  $\text{Al}_2\text{O}_3$  content increases its mechanical strength and hardness, as well as thermal expansion coefficient.

Oxidic ceramics have high strength, hardness, corrosion resistance, and they are very good electric insulators. They are used mainly at the machining of cast iron, as a structural material for components of high-temperature devices, melting crucibles, cutting plates for metals machining, spark plug insulators for engines, etc.

## 2.2 Non-oxidic ceramics

This group includes mainly nitrides, carbides and borides. Non-oxidic ceramic materials have higher strength at high temperatures in comparison with oxidic ceramics. They have higher heat conductivity and lower thermal expansion coefficient than oxides, which means they have a better resistance to thermal shocks. For this reason they are in practice used mainly as high-temperature structural materials. Their disadvantage consists in lower resistance to oxidation. In non-oxidic compounds covalent bond prevails (for example SiC, B<sub>4</sub>C, Si<sub>3</sub>N<sub>4</sub>, BN) or metallic bond (for example TiC, UC, WC, TaC, TiN). Carbides and nitrides have the broadest use of non-oxidic ceramics, mainly in special fields of mechanical and electrical engineering.

## 2.3 Mixed ceramics

It is formed by a combination of structural oxidic and non-oxidic ceramics. Composite cutting tools based on Al<sub>2</sub>O<sub>3</sub>+ZrO<sub>2</sub>+SiC are used for the lathe work of quenched steels and hardened cast irons, at the end of the machining process, such as milling. Except SiC, the most frequently used non-oxide additives are TiC or TiN.

## 3. Ceramic properties

The physical, mechanical and chemical properties of ceramic materials depend on their chemical composition and micro-structure.

### 3.1 The positive properties of ceramic materials are the following:

- low density
- low thermal expansion coefficient
- high hardness
- high temperature and corrosion resistance
- high wear resistance
- high melting temperature
- stability of mechanical properties in a wide temperature interval

### 3.2 Main disadvantages of ceramic materials are the following:

- brittleness
- difficult reproducibility of complex shapes
- highly demanding processing
- difficult joining of different ceramic components or between ceramic components and other materials

### 3.3 Causes of brittleness

Brittleness is the main problem of structural ceramics. It expresses itself by:

- the creation of brittle failure
- static fatigue
- the degradation of properties at mechanical and thermal shocks

It results from the constitution of ceramic materials, that it is suppressed in them the development of plastic deformation under conventional conditions.

The causes of brittleness are divided into:

- Fundamental
- Secondary

FUNDAMENTAL CAUSES result from the type of inter-atomic bond (physical cause), but also from the type of crystalline lattice (geometric cause).

Fundamental causes of brittleness are essentially caused by **low mobility of dislocations** and by **bad manoeuvrability**.

Low mobility of dislocations is related to the high level of the Peierls–Nabarro stress in materials with an ionic and covalent bond. Bad manoeuvrability is connected with non-fulfilment of the requirement of five independent slip systems in single crystals – so called von Mises criteria.

The fundamental causes of ceramic brittleness as a result of ionic and covalent bonds comprise also a less perfect connection of the lattices along the grain boundaries, than with metallic bonds which manifest itself by a lower cohesion strength on the grain boundaries.

Brittleness transition temperature  $T_P$  exists in ceramic materials. Below this temperature the materials suffer brittle failure – they are unable to deform plastically. The brittleness is caused by the fact that a plastic zone is not formed in front of initiated cracks and the crack propagates by cleavage along the grain boundaries or through the extend of the grains.

Fundamental causes do not lead to absolute suppression of macroscopic plastic deformation below the transition temperature  $T_P$ . For example, BeO, AlN,  $Al_2O_3$  in a poly-crystalline state at superimposed multiple pressures are capable even at room temperature of a plastic deformation up to 10% - easier formability of ceramics while loading with a big hydrostatic pressure. The strength of the boundaries is sufficient for the movement of dislocations and the activation of additional slip planes while realizing shear stress.

SECONDARY CAUSES include the heterogeneity of the structure and properties, as well as the significant weakening of the grain boundaries due to an imperfect sintering and to the presence of pores, cracks and vitreous phases along grain boundaries. The characteristic defects of ceramics include cracks and other defects caused by incorrect technological process at forming, drying or sintering.

The level of mutual contact of crystalline grains or of their contact with vitreous or binding phase is of great importance. The pores significantly influence the strength of ceramics by reducing the carrying section, as well as by the fact that they act as stress concentrators.

The mentioned secondary causes of brittleness are usual sources of failure of traditional ceramics already at low stresses. They are partly suppressed at structural ceramics.

These secondary causes are strongly applied at high temperatures, when the softening of the vitreous phase occurs and there is an easy slip along the grain boundaries, which cannot be compensated by the plastic deformation of the grain. It leads to the initiation of cracks and to their brittle inter-crystalline propagation above the transition temperature  $T_P$ .

#### 4. Possibilities to enhance the mechanical properties by toughening the ceramics

Minimum capability of plastic deformation has an essential influence on the achieved level of mechanical properties of structural ceramics. The enhancement of the level of the mechanical properties can be therefore achieved by increasing the toughness of the ceramic materials, i.e. by the achievement of a certain level of development of the process zone for

the cracks under limited conditions. The increase of the fracture toughness can be achieved by several ways as follows.

#### 4.1 Structure optimization

In this possibility to increase the toughness we talk about the so-called engineering of the grain boundaries. It consists in the reduction of porosity, the removal or reduction of the volumetric part of the vitreous phase on grain boundaries. This reduction requires highly pure initial powders and the perfection of all of the production of ceramics in sintering and pre-sintering processes.

If the ceramic contains a large portion of the vitreous phase, the crack propagates mainly through this phase and other phases have very little influence on this propagation. Ceramics with increased fracture toughness should contain only a very small part of the vitreous phase.

Another condition is a homogenous fine-grained material, a grain size from tenths of micrometers to units of micrometers. The pores of spherical shape, occurring for example in the vitreous phase, can even have a favourable influence, since they obstruct the front of the crack.

The shape of the grains and their way of strengthening is of great importance. For example  $\text{Si}_3\text{N}_4$  with **acicular grains** has even **4-times higher** fracture toughness -  $K_{IC}$  - than in the case of **uniform equi-axial grains** (Jonšta, 1998).

Increase the strength (toughening), i.e. to increase of resistance to crack propagation, can be achieved with the effect of dispersive particles. At the use of these particles the solid grains of the dispersed phase cause a deviation of the crack from its direct course, and this results in an extension of the fracture area. Except that the particles take over a greater part of the load and they thus reduce the stress on the matrix. This final result leads to a reduction of the stress factor on the front of the crack.

#### 4.2 Phase transformation

It is based on an induced phase transformation of the dispersed particles in the matrix of a ceramic material. It is used mainly in the area in front of the crack, where the effectuation of a **martensitic phase transformation** contributes to energy absorption. A pre-requisite for using this type of strengthening is the requirement that the transforming material must have a greater volume of its low-temperature modification than it has in the high-temperature structural modification.

The considered process must therefore lead to the creation of a phase with bigger volume, where the phase transformation should be made with a martensitic mechanism. It may also be assumed that the strains are not relaxed, since the relaxation process proceeds very slowly. Among the materials that have this behaviour, zirconium dioxide ( $\text{ZrO}_2$ ) seems to be the most advantageous technical ceramic.

$\text{ZrO}_2$  crystallizes in three modifications:

- \* *cubic* - stable up to 2680°C
- \* *tetragonal* - stable up to 2370°C
- \* *monoclinic* - stable up to 950°C

A big volumetric change at the transition of tetragonal modification to a monoclinic one, when relaxation by diffusion or relaxation mechanisms is impossible, is the cause of the fact that pure  $\text{ZrO}_2$  cannot be used as ceramic. That is because at cooling from the sintering temperature it could crack along the grain boundaries, at the modification change temperature, due to big internal strains.

If we stabilize this modification till room temperature (so called complete stabilization) by the addition of various oxides that are soluble in cubic modification of  $ZrO_2$ , such as for example  $Y_2O_3$ , in such case the detrimental transition from tetragonal to monoclinic modification cannot occur in whole volume.

The transformation of tetragonal modification to a monoclinic one occurs only at the places of high tensile stresses, i.e. in the front of the crack. Due to the fact that the monoclinic phase has a volume approximately by 4% bigger than the tetragonal phase, the development of pressure strains occurs at these places, which modify the state of stress on the crack and thus slows a further development of cracks.

The following three mechanisms participate in a decisive way on energy dissipation at a material failure, initiated by strengthening at phase transformation:

- stress induced transformation in the course of load
- initiation of fine cracks
- deviation of cracks from their initial direction of growth

The stress induced martensitic phase transformation of tetragonal  $ZrO_2$  to monoclinic is significantly manifested by a *protective effect of the crack*, which is accompanied by the above mentioned *volume changes*. The dissipation of fracture energy occurs by the energy consumed for the nucleation of phase transformation and the energy consumed for the creation of a new volume originated by transformation. As a result of the already mentioned increase of volume, the tensile stresses are also eliminated (Pokluda et al., 1994).

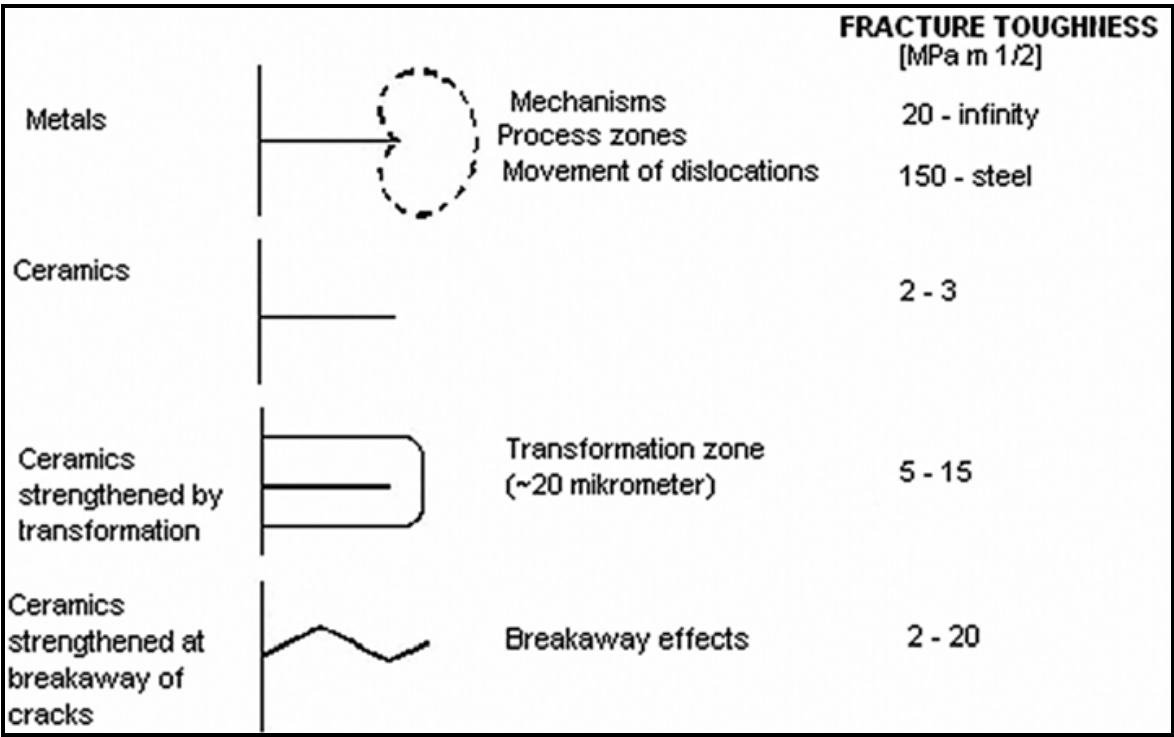


Fig. 1. Schematic representation of the effects related to the dissipation of energy at crack growth in metallic and ceramic matrices

The creation of fine micro-cracks in the regions of dispersed particles, in which the tetragonal phase has transformed into a monoclinic phase, has a favourable effect on the increase of the fracture toughness. These cracks have usually lengths of 10 to 100 nm. They are initiated around the front of the crack in the process zones. Their frequency in the matrix

can be quite high and at the increase of the load there can be appropriately oriented micro-cracks expanded by a certain part of their original length, which is also connected with their ramification.

As a result we have the reduction or elimination of local stress peaks. It is possible to imagine this process as the distribution of stresses induced on the front of the crack onto a large number of small cracks. The material behaves under these conditions in an elastic-plastic manner, because the opening of micro-cracks is at the unstressing, partly reversible.

#### 4.3 Formation of composite materials

The biggest increase of the resistance to crack propagation, i.e. the increase of the level of ceramic's fracture toughness, promises the use of compound ceramic materials - composites. This means using the strengthening effect of ceramics thin fibres or whiskers in the ceramic matrix. The diameter of the used fibres is usually several  $\mu\text{m}$  and their length is of several millimetres, while the whiskers' dimensions are smaller almost by one order. Optimum properties are achieved, if the cracks do not propagate through the fibres, and a considerable part of energy is consumed on extraction of the fibres and at the failure of the fibre interface - matrix.

### 5. Evaluation of mechanical properties of structural ceramics

The basic characteristics of structural ceramics are the fracture toughness, tensile and bending strength, compressive strength, elastic constants, the parameters of the statistical distribution of strength and parameters of growth curves of the statistical or dynamic fatigue. The evaluation of these parameters is often difficult, because there are a number of empirical relationships for the characterization of ceramic materials.

Right now, the attention is devoted mainly to the determination of the fracture toughness by the use of the indentation technique. This technique, usually used to measure hardness, finds its application in the evaluation of these important material characteristics. In our case it was used the Vickers indenter. In the use of these techniques there are measured the length of the cracks initiated at the corners of the indentation print when it is exceeded the critical value of the loading force, which involves the initiation of cracks and their subsequent development as shown in Fig. 2.

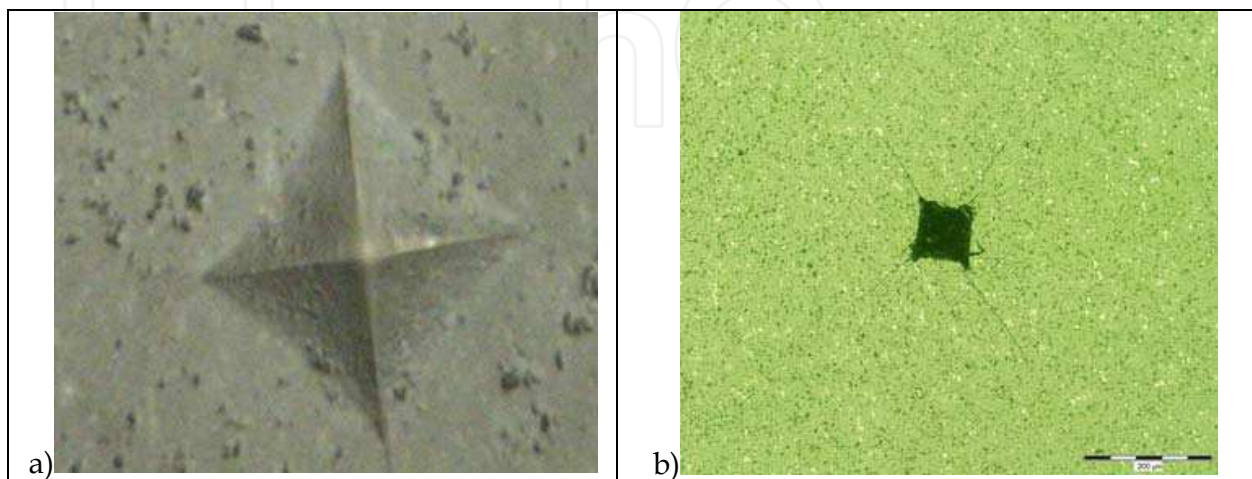


Fig. 2. Vickers' print with a load of 10 N, for a sample material a) zoom 1000x b) zoom 100x

This approach, however, requires overcoming some limiting characteristics which are related to the type of the initiated cracks. Preferential incidence of one type or another is related to the type of materials and the intensity of load on the print. On Fig. 3 are schematically illustrated the cross-section variations of the initiated cracks by Vickers indentation. This is known as radial / median crack (see Fig. 3a), where  $m_t$  corresponds to the median crack and  $l_t$  to the side (lateral) crack. The figure 3b shows the radial or Palmquist cracks  $r_t$  and  $l_t$  shows the side (lateral) cracks. The Palmquist cracks type can be observed at lower levels of indenter loads and on the contrary the radial / median cracks are initiated at higher levels.

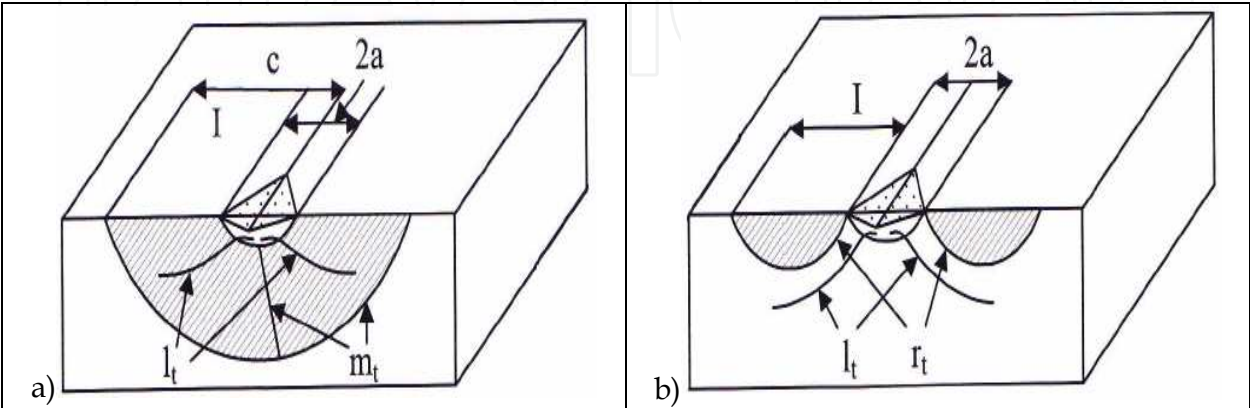


Fig. 3. a) median crack, b) lateral crack

This evaluation technique requires the preparation of a relatively small area of the material sample for the indentation prints. Of course, there a number of restrictive conditions in its application related both to the level of surface preparation, and to the conditions for the initiation and propagation of the cracks. We could also consider a risk on the evaluation associated with the use of small areas and the fact that the measurement is performed on the material surface (assuming that the homogeneity and the volume of the tested material is maintained).

Matzke pointed out these limiting factors, on behalf of his special attention to the study of this kind of situation (Matzke, 1980, 1990, 1991). It should also be taken into account the possible presence of discontinuities by major or minor deviations in the width of cracks. It cannot be excluded in some cases the possibility to initiate more than one indentation crack in the corners of the print.

According to Matzke this leads to a possibility of underestimation of the crack length, but in macroscopic terms the fracture toughness values would be realistic data (Matzke, 1991). The processes of uneven development of cracks are an integral part of a complete evaluation of the parameter of resistance to brittle fracture.

In our works we have used, for determining the values of fracture toughness, a relationship that is independent of the crack profile and allows you to evaluate different types of materials or a type of material in a wide range of applied loads. This relationship was presented by Liang et al. (in 1990) and is expressed as follows:

$$K_{IC} = \frac{\left[ \frac{2l}{u} \right]^{\left( \frac{l}{9u} - 1,51 \right)} \cdot HV \left( \frac{u}{2} \right)^{\frac{1}{2}}}{\left( \frac{HV}{E \cdot \Phi} \right)^{0,4} \cdot \alpha \cdot \Phi} \quad [\text{MPa m}^{1/2}] \tag{1}$$

with:

$$\alpha = 14 \left[ 1 - 8 \left( \frac{4\nu - 0,5}{1 + \nu} \right)^4 \right] \quad (2)$$

and the length of the crack is:

$$l = \frac{\sum_{i=1}^n l_i}{4} \quad [\text{m}] \quad (3)$$

Where:

HV – hardness by Vickers,

u – diagonal length,

l – crack length,

$\alpha$  – dimensionless constant dependent on the order of the Poisson's number of a particular type of ceramic material – 9.3,

$\Phi$  – limiting factor – 3,

E – Young's module of elasticity.

With the indentation technique we can make an evaluation of structural ceramics response to repeated loadings. Here, we also have similar limitations as in the previous case. Informatively it may be noted that in the evaluation of the kinetics the advances of the fatigue cracks in structural ceramics the obtained and modified characteristics are compared with the results obtained at structural steels. Although this technique does not allow to establish an analogical relation with Paris' kinetic equation, it allows to determine the response of a certain type of ceramic to repeated loading based on the dependence between the number of burdened cycles and the length of the induced cracks, or in that case based on a defined number of burdened cycles leading to the chip of the evaluated material around the indentation print.

## 6. Indentation model of the origin of cracks

At the formation of a Vickers' indentation print an elastic-plastic stress fields in the material are originated, which affects the subsequent expansion of cracks. The period of the cracks derivation is an important factor that determines the fracture threshold value and could be the control parameter for determining the dominant influence of the surface radial cracks or of the subsurface median cracks in the first stage of propagation.

According to the model of Marshall and Lawn, the fully loaded indenter with an elastic / plastic field is considered as a superposition of a residual field of an unloaded material and as an ideal plastic contact field. The essence of the mentioned model is the assumption that during the unloading operation of the indenter does not appears the action of a reverse plasticity. (Marshall & Lawn, 1979)

During the indenter loading, in the material the origin of elastic deformation zones occur with the subsequent development of the radial / median cracks. The growing tensions cause the subsequent stable growth of median cracks. During the subsequent unloading the closure of median cracks occurs, while the lateral cracks grow until the indenter load is completely removed, and there is when the subsequent chipping can occur (Cook & Pharr,

1990). The initiation and propagation of the radial/median and lateral cracks leads, in brittle materials, to a subsequent chipping formation. On Fig.4 a schematic representation of the cracks system under Vickers' indentation print is shown.

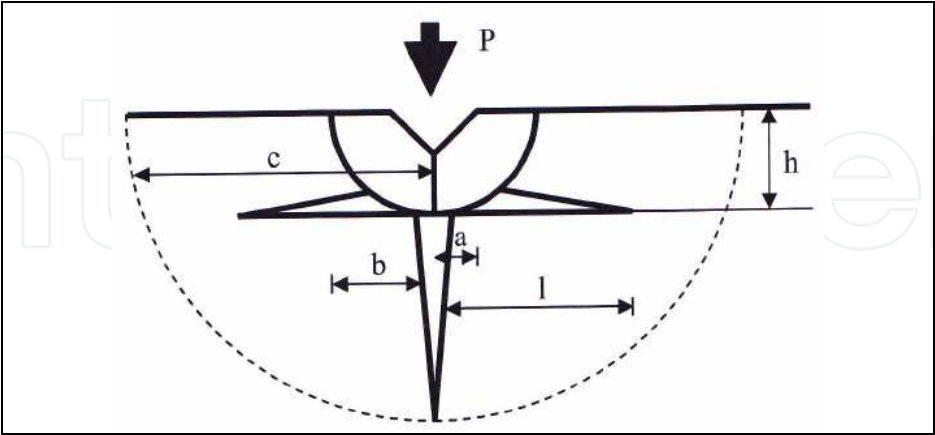


Fig. 4. Schematic representation of the crack system under Vickers' print

On the presented picture - P- is the index load at which the origin of median cracks with a length of - c - occurs. The plastic zone is formed during the application of the Vickers' load of the indenter with an appropriate angle  $2\psi$ , where - a - is half the diagonal of the Vickers' print and - b - is the plastic deformation zone accommodated under the print in the plastic zones. The critical stress for the formation of radial/median crack is given by (Evans & Marshall, 1981; Kumar & Khamba, 2006):

$$P = \alpha \cdot \frac{K_{IC}^4}{HV^3} \quad [N] \tag{4}$$

where:  
 $\alpha$  - dimensionless constant  
 $K_{IC}$  - fracture toughness  
HV - hardness by Vickers

At the Vickers indenter loading, just below the indentation print in the material, the formation of irreversible strains occurs. From the schematic view of the cracks resulting in a material when exposed to Vickers indentation, we have that  $\delta V$  is the printed volume, V is the volume of the deformed zone and  $\psi$  is half the size of the Vickers' indentation print. Then the next equation applies:

$$\frac{\delta V}{V} = \left(\frac{a}{b}\right)^3 \cdot \cot g\psi \tag{5}$$

This zone is to maintain its constant volume elastically compressed with the surrounding material. The result is the formation of a residual force  $F_r$  on the surface of the indentation print, which then acts on the axis of the indenter in the opposite load direction. At the same time the formation of the residual forces  $F_m$  occurs, which are located in close proximity to the plastic zone. These residual forces cause on their surroundings the creation of tensile stresses and with the absence of a reverse plasticity they achieve, at full loading of the indenter, the maximum levels of the force F ( $F_{r0}$  and  $F_{m0}$ ).

This level is maintained even after the subsequent unload of the indenter, if during the loading there are no material cracks. At a sufficient level of strength of  $F_{r0}$  occurs after unloading the indenter the formation of lateral cracks at the depth  $h$ , while the power  $F_{m0}$  is associated with the formation of radial/median cracks. The lateral crack grows up to the equilibrium length  $l$  and the residual strength, leading to the formation of these cracks, is reduced from the maximum level  $F_{r0}$  to the level equilibrium value  $F_r$  (Evans & Marshall, 1981).

Marshall et al. (in 1982) derived an equation from an elastic-plastic analysis that expresses the relationship between the so-called equilibrium length of the lateral cracks  $l$  and the index load:

$$l = l_0 \cdot \left[ 1 - \left( \frac{F_0}{F} \right)^{\frac{1}{4}} \right]^{\frac{1}{2}} \quad [\text{mm}] \quad (6)$$

where  $F_0$  is the apparent threshold value, which can be expressed with the use of the fracture toughness:

$$F_0 = \frac{\zeta_0 \cdot \frac{E}{H} \cdot K_C^4}{A^2 \cdot (\cot g\psi)^{\frac{2}{3}} \cdot HV^3} \quad [\text{N}] \quad (7)$$

where  $l_0$  corresponds to the length of the crack limits defined by the following equation:

$$l_0 = \left[ \frac{\zeta_l \cdot (\cot g\psi)^{\frac{5}{6}} \cdot \left( \frac{E}{H} \right)^{\frac{3}{4}}}{A^{\frac{1}{2}} \cdot K_C \cdot HV^{\frac{1}{4}}} \right] \cdot F^{\frac{5}{8}} \quad [\text{mm}] \quad (8)$$

In this equation  $\zeta_0$  and  $\zeta_L$  are dimensionless constants independent of the system material - indenter. These constants determined Marshall et.al. (in 1982), based on the measurements of ceramic materials. The constant  $\zeta_0$  corresponds to the value  $1,2 \cdot 10^3$  and the constant  $\zeta_L$  to the value  $25 \cdot 10^{-3}$ . The magnitude  $A$  on the previous equations is a dimensionless geometric constant. Using the previous relationships it can be predicted the dependence between the equilibrium length of lateral cracks and indenter's load.

## 7. Crack propagation and chipping formation

During the loading, in the material occurs the crack propagation and may subsequently also occur the chipping formation. The formation of cracks in the index corresponds to the time of contact of the indenter with the surface of the evaluated material and its subsequent entry into the volume of the material.

This process can be simply described as a penetrating process that leads to plastically deformed zones surrounding the Vickers' indentation prints. There also occurs the formation of the surface tension zones which are adjacent to the contact places between the indenter and the material and occurs the emergence of a field for tensile stresses in the front of the indenter (Antis et al., 1981).

The perforation of the indenter into the sample surface leads to the initiation of subsurface cracks at the indentation's front. At this place the subsurface tensile stress levels reach their maximum. A bigger load leads to the development of already initiated median type cracks, in both directions: towards the material and towards the surface of the sample - radial/median crack- (see Fig.5).

The median crack remains, in the case of a no sufficient load during the loaded cycle, under the surface of the evaluated material, which means that it is primarily propagated into the volume of the material and only a minority to its surface.

The subsequent intersection of the cracks with the surface of the sample can be detected only after unloading, when the active surface compressive stresses are sufficiently reduced (Sglavo & Green, 1995).

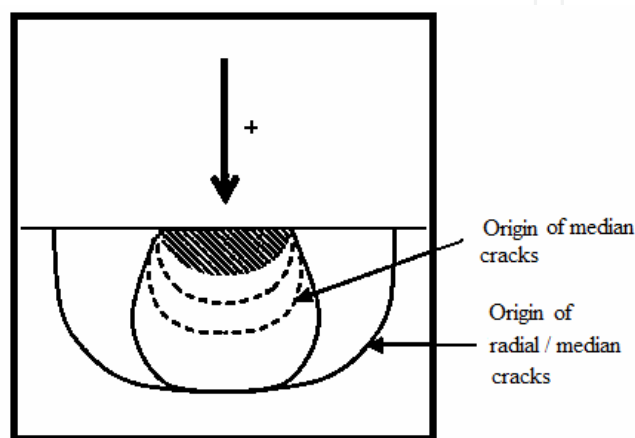


Fig. 5. Schematic representation of the origin of median and radial/median cracks under load

The median crack has a tendency to close during the subsequent unload if the indenter. The full closure will not result because of the action of the fields of internal stresses, which is related to the execution of the elastic-plastic state. Such induced residual stress fields cause the formation of lateral (side) cracks that are initiated at the plastic zone and then propagated in a parallel to the sample surface plane. The development of lateral cracks continues during the indenter's unloading.

The initiated induced internal stresses affect the next development of subsurface median or eventually radial/median cracks. Dissemination of lateral cracks increases proportionally with increasing the load of the indenter. Their intersection with the surface of the material leads to the chipping formation.

## 8. Composite materials based on ceramics

As previously mentioned, composite (compound) materials present due to their high resistance to brittle crack propagation, that is because of their high fracture toughness, promising materials for technical applications. Particularly, ceramic nanocomposites meet the demands of modern technologies.

Micro / nanocomposites based on nitride and silicon carbide are a new generation of the ceramic materials developed in the last decade. The range of materials based on  $\text{Si}_3\text{N}_4$  is recently widely used in the manufacture of cutting tools, for components exposed to friction, combustion engine components, water pumps, industrial pumps, piston engines, gas turbines etc. This type of ceramic material is characterized mainly by the high strength

at normal and elevated temperatures, chemical stability, good hardness, wear resistance, low coefficients of friction and low density.

Ceramics and ceramic composites are nowadays used in various technological fields. The ceramic composites are classified according to their particles size into microcomposites where the secondary phase particle size is measured in microns, and nanocomposites where there is a nanometric size of the secondary phase particles.

In addition to improved mechanical properties of ceramic nanocomposites there were observed new features such as machinability and superplasticity. The aim of the preparation of ceramic composites is to improve not only the "traditional" mechanical properties such as hardness, strength and reliability at room temperature, but mainly to improve the mechanical properties at high temperatures, such as creep resistance to high temperature, strength and corrosion resistance.

As a disadvantage we can see quite a significant financial demand of the preparation of nanocomposites because of the high prices of the nanopowders and the expensive furnace equipment, because the compaction of the nanocomposite requires special methods of sintering with rapid heating and short holding time at high temperature.

The ceramic nanocomposites can be divided into four categories (Fig. 6): *intragranular nanocomposites*, *intergranular nanocomposites*, *intra-intergranular nanocomposites* and *nano / nano composites*. Intra and intergranular nanocomposites have a two to five times higher toughness and strength at room temperature than the monolithic materials.

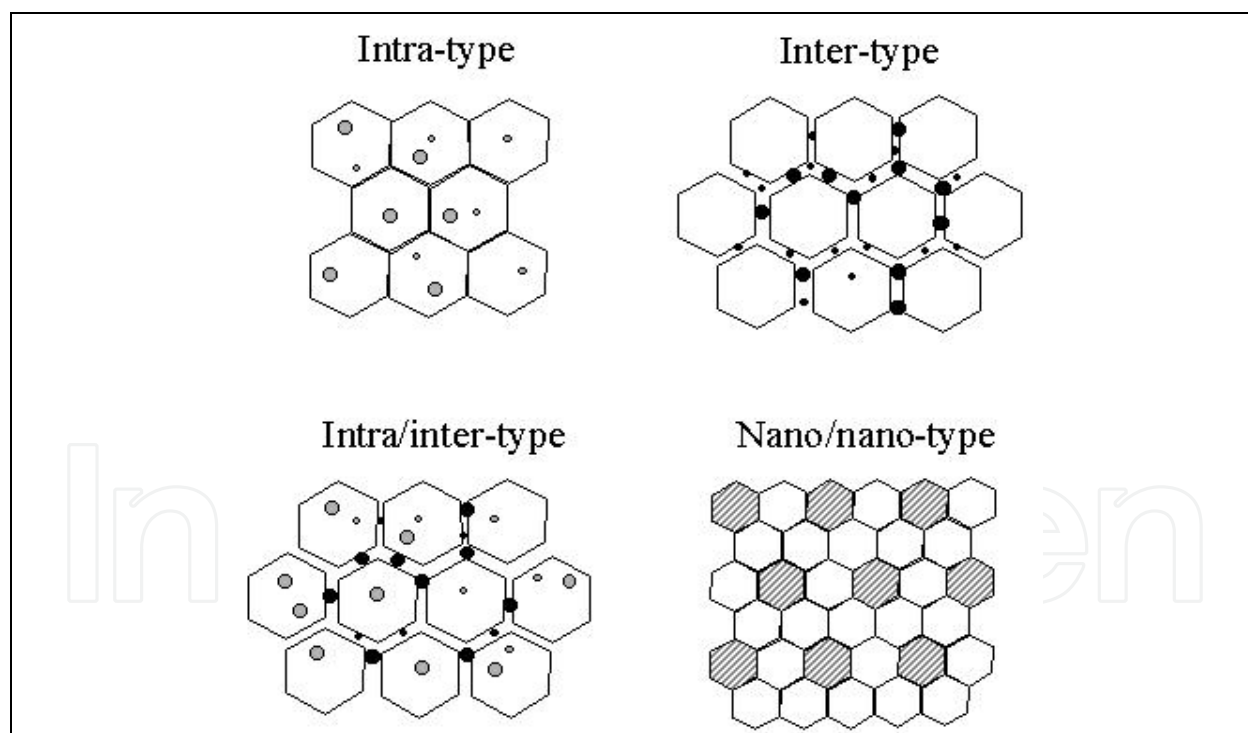


Fig. 6. Types of ceramic nanocomposites (Jonšta et al., 2010)

## 9. Nanocomposites based on $\text{Si}_3\text{N}_4/\text{SiC}$

Ceramic materials on the base of  $\text{Si}_3\text{N}_4$  are recently one of the best candidates for tribological application due to the combination of their good thermal and mechanical properties, chemical resistance and high hardness.

On recent investigations (Bolaños et al., 2010) it was studied the preparation of Si<sub>3</sub>N<sub>4</sub>/SiC nanocomposites with different rare earth oxide sintering aids (La<sub>2</sub>O<sub>3</sub>, Sm<sub>2</sub>O<sub>3</sub>, Yb<sub>2</sub>O<sub>3</sub> a Lu<sub>2</sub>O<sub>3</sub>) and the evaluation of their hardness, indentation fracture toughness and wear resistance at room temperature.

9.1 Analyzed results of the investigation

9.1.1 Density

The density of the samples after the hot pressing process amounted to 99%, so it is considered that the sintering conditions were chosen properly. At temperatures above 1510° C, there was a decrease in the vacuum, ie. CO gas was liberated proving the formation of SiC with the carbothermic reduction. After this reaction and the subsequent increase on the temperature to 1650° C occurred the formation of the liquid phase in the system RE<sub>2</sub>O<sub>3</sub> - SiO<sub>2</sub>. In the presence of Si<sub>3</sub>N<sub>4</sub>, the ternary eutectic point is lower, as for binary systems. The formation of liquid phase allowed the compaction of the ceramic composite. The densities of the sintered samples prepared by hot pressing are shown in table 1.

Sample	Density ρ [g·cm <sup>-3</sup> ]
K3Lu-2	3,378
K3Yb-2	3,348
K3Sm-2	3,322
K3La -2	3,289

Table 1. Densities of the K3Re-2 nanocomposite materials

9.1.2 Microstructure

The microstructure of the hot-pressed composite materials denominated as K3La, K3Sm, K3Yb and K3Lu are shown in Fig. 7 and Fig. 8. According to the observed microstructures, we can conclude that the samples are slightly over etched (Si<sub>3</sub>N<sub>4</sub> fine particles below 300 nm are not visible), so visually it appears that glass phase is more than 10% by volume. In all samples the microstructure contains large elongated particles of β-Si<sub>3</sub>N<sub>4</sub> (whiskers) which are surrounded by finer particles of β-Si<sub>3</sub>N<sub>4</sub> with needle shapes. We call this fine fraction the matrix and we call the larger β-Si<sub>3</sub>N<sub>4</sub> whiskers the tougher particles.

It was expected a different evolution of the microstructures because of the used oxide RE<sub>2</sub>O<sub>3</sub>. A more detailed study of the Fig. 7 and Fig. 8, shows that the microstructure of the sample matrix of K3Yb and K3Lu is fine-grained. The large extended β-Si<sub>3</sub>N<sub>4</sub> grains are comparable in thickness with the sample K3La, which has lower viscosity of the glass phase. In the process of sintering of Si<sub>3</sub>N<sub>4</sub>, some α-Si<sub>3</sub>N<sub>4</sub> grains of the primary powder dissolved in the liquid phase and then precipitated, and grew in the form of β-Si<sub>3</sub>N<sub>4</sub> needles.

Their growth was mainly controlled by diffusion, and that is why, in systems with a higher viscosity of the liquid phase (system containing Yb and Lu) the transport of material from the melting α-Si<sub>3</sub>N<sub>4</sub> grains to the growing β-Si<sub>3</sub>N<sub>4</sub> grains was slower, and that means that the microstructure of the matrix of these materials has then finer grains.

Another possible reason for the fine-grained microstructure of the systems with higher viscosity, is the slower dissolution of the original β-Si<sub>3</sub>N<sub>4</sub> grains (about 5 wt.% on the

starting  $\text{Si}_3\text{N}_4$  powder SN-E10) with small diameter so-called critical diameter (0,1 to 0,2 $\mu\text{m}$ ). On the systems with the addition of  $\text{La}_2\text{O}_3$  and  $\text{Sm}_2\text{O}_3$  the dissolving speed of the  $\text{Si}_3\text{N}_4$  is slightly higher, and therefore the matrix of these systems is slightly coarse-grained.

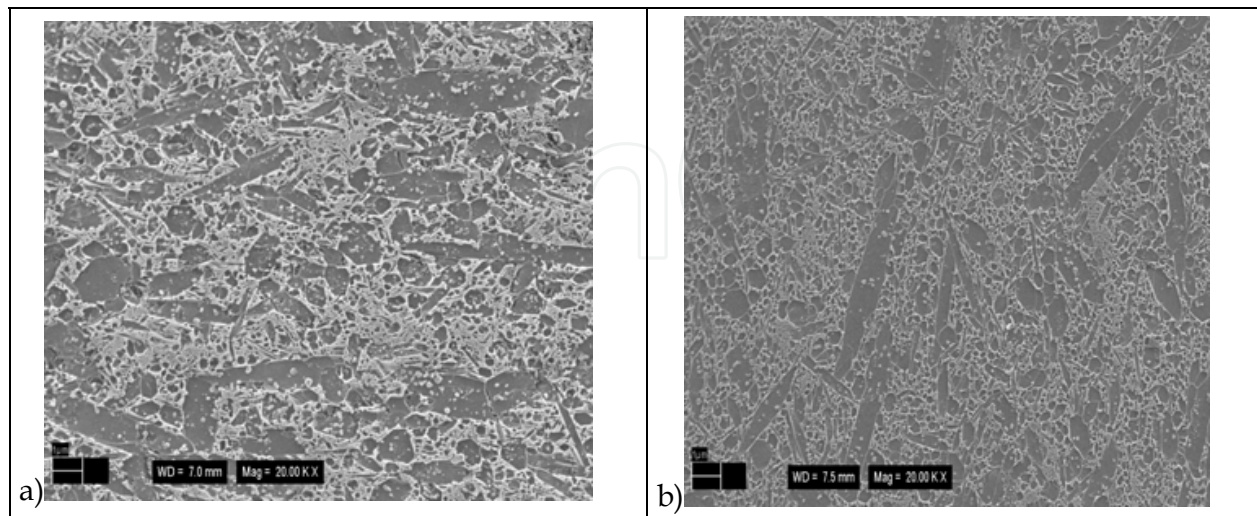


Fig. 7. Microstructure of samples a) K3La, b) K3Sm obtained by SEM at 20000<sup>th</sup> magn

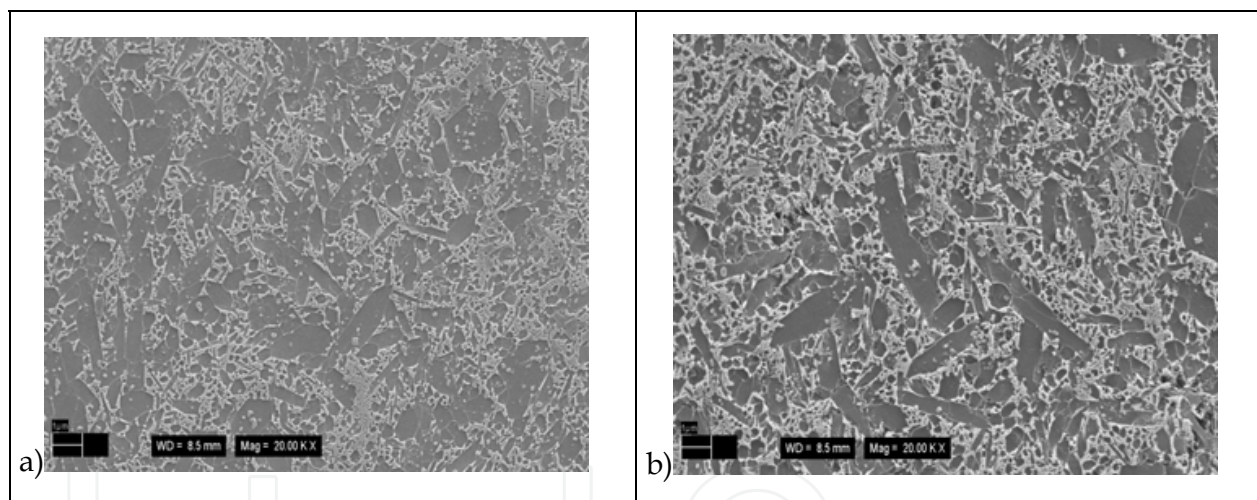


Fig. 8. Microstructure of samples a) K3Yb, b) K3Lu obtained by SEM at 20000<sup>th</sup> magn

The rare earth metal oxides also affect the thinness of the  $\beta\text{-Si}_3\text{N}_4$  particles. The average value of slenderness decreases with the growing ionic radius of the rare earth metals, or with the decrease of the viscosity of the liquid phase.

The growth of elongated  $\beta\text{-Si}_3\text{N}_4$  grains is affected not only by the viscosity of the liquid phase, but also by the ability of rare earth elements to adsorb itself on the surface of  $\text{Si}_3\text{N}_4$  crystals. It was observed, with a transmission electron microscopy, that Lu has a higher stability in an oxy-nitride glass (stronger bond with O), while La adsorbs preferentially on the prismatic surface of the  $\text{Si}_3\text{N}_4$  grains (binding with N), thereby preventing the further build up of dissolved Si in the surface of N, and thus growing on the thickness of the  $\beta\text{-Si}_3\text{N}_4$  grains.

From the observation of 15 photomicrographs of each sample of K3RE-2, was showed that with the decrease of the ionic radius of the rare earth elements ( $\text{La} \rightarrow \text{Sm} \rightarrow \text{Yb} \rightarrow \text{Lu}$ ) the structure is more bimodal, the matrix is finer and therefore has more elongated particles of

$\beta$ - $\text{Si}_3\text{N}_4$ . From La to Lu their thickness slightly increases. For a more detailed statistical analysis it is necessary to measure the slenderness of at least 2000 grains of each microstructure of each ceramic material containing K3RE.

The microstructure of the samples contains, together with elongated  $\text{Si}_3\text{N}_4$  grains, irregularly distributed SiC particles, which are located both at grain boundaries and inside the  $\text{Si}_3\text{N}_4$  grains. The larger SiC particles are mainly located at the grain boundaries (inter SiC grains), and the finer SiC particles (<50 nm) were observed within the  $\text{Si}_3\text{N}_4$  grains (intra SiC grains). The SiC nanoparticles can slow up, or may completely stop the abnormal growth of the  $\beta$ - $\text{Si}_3\text{N}_4$  grains. Therefore, the overall microstructure of the composites with K3RE was finer compared with the reference samples.

### 9.1.3 Abrasion resistance

The abrasion resistance tests were performed using the "ball-on-disc" method. The friction curve had a similar pattern for all the tested samples (K3RE-1). At the beginning, the friction coefficient was low, but then increased to a maximum value and then stabilized at a relatively constant value during the test.

The Fig.9 shows the friction coefficients of reference (monolith:  $\text{Si}_3\text{N}_4 + 3 \text{ mol. \% RE}_2\text{O}_3$ ) and composite samples with K3RE ( $\text{Si}_3\text{N}_4 + \text{SiC} + 3 \text{ mol. \% RE}_2\text{O}_3$ ). The friction coefficient of the monolithic samples was higher compared with the composite samples with the same additive  $\text{RE}_2\text{O}_3$  (RE = La, Sm, Yb, Lu). The lower friction coefficient of composite materials is attributable to their higher hardness compared to the reference monoliths and the presence of SiC nanoparticles. From the figure 3 it is also clear, that the friction coefficient slightly decreases with decreasing the ionic radius of  $\text{RE}^{3+}$ . The better sliding properties of the composites with K3Lu and K3Yb compared with K3Sm and K3La may be due to a finer microstructure of the matrix (Fig. 7-8) and to a higher rigidity of the glassy phase at the grain boundaries, because the glass with a smaller radius of the  $\text{RE}^{3+}$  ion has a higher hardness, fracture toughness and Young's modulus.

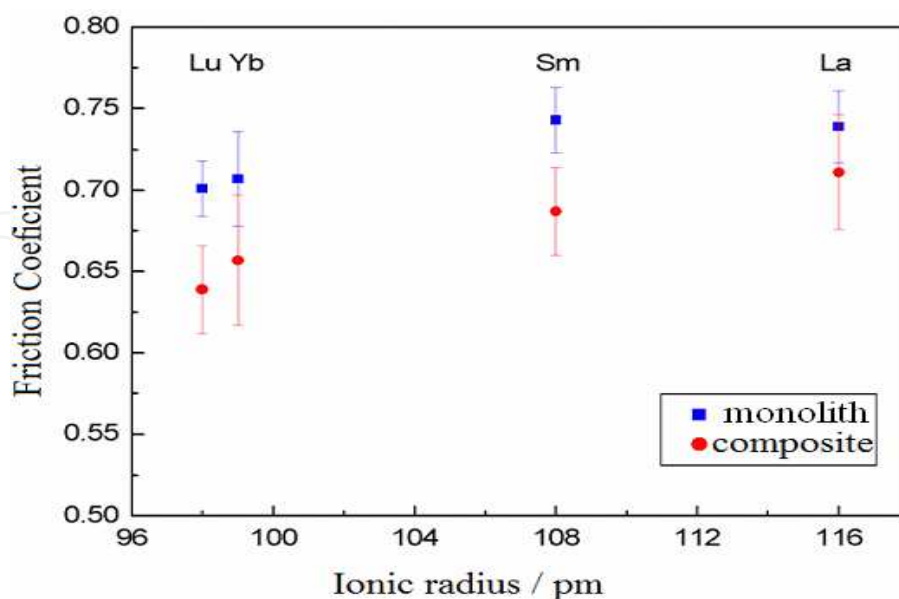


Fig. 9. Effect of ionic radius of  $\text{RE}^{3+}$  sintering additives on the friction coefficients of the reference monoliths ( $\text{Si}_3\text{N}_4 + 3 \text{ mol. \% RE}_2\text{O}_3$ ) and composite materials K3RE ( $\text{Si}_3\text{N}_4 + \text{SiC} + 3 \text{ mol. \% RE}_2\text{O}_3$ )

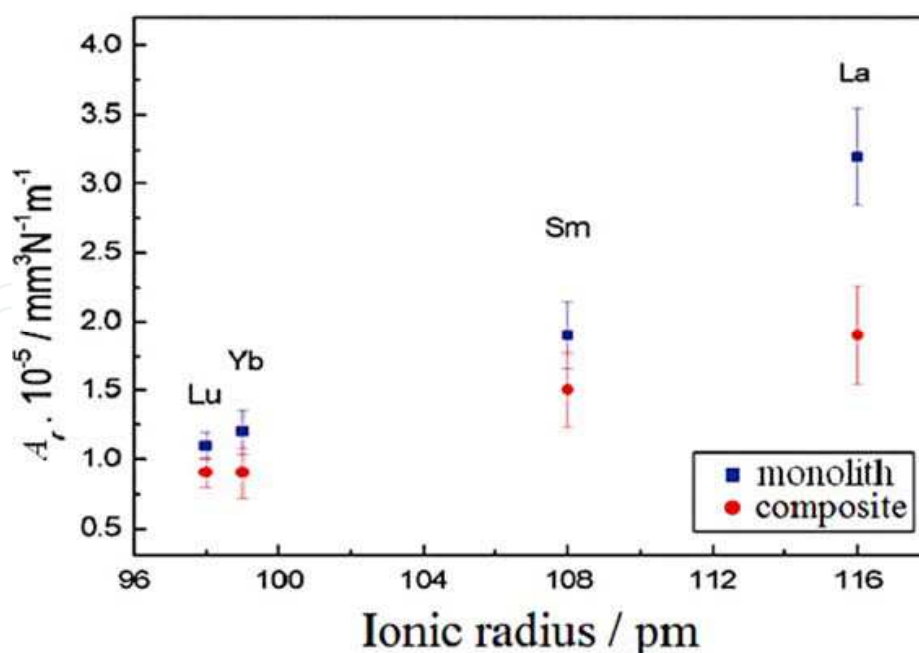


Fig. 10. Effect of ionic radius of RE<sup>3+</sup> sintering additives on the abrasion rate ( $A_r$ ) of the reference monoliths ( $\text{Si}_3\text{N}_4 + 3 \text{ mol. \% RE}_2\text{O}_3$ ) and composite materials K3RE ( $\text{Si}_3\text{N}_4 + \text{SiC} + 3 \text{ mol. \% RE}_2\text{O}_3$ )

The Fig. 10 shows the relationship of the abrasion rate ( $A_r$ ) of the reference monoliths ( $\text{Si}_3\text{N}_4 + 3 \text{ mol. \% RE}_2\text{O}_3$ ) and the composite materials K3RE ( $\text{Si}_3\text{N}_4 + \text{SiC} + 3 \text{ mol. \% RE}_2\text{O}_3$ ) with the ionic radius of the RE<sup>3+</sup> sintering additives. The difference in the abrasion rates is more pronounced than the difference in the friction coefficients; however, the tendency is maintained. The samples with the addition of smaller RE<sup>3+</sup> ionic radius (K3Lu and K3Yb) exhibit less abrasion in comparison with the samples containing additives with larger ionic radius (K3Sm and K3La). This is expected to be due to a higher rigidity of the glassy phase of the samples K3Lu and K3Yb because, in general, the abrasion rate of the ceramic materials based on  $\text{Si}_3\text{N}_4$  with lower strength on the grain boundaries is higher. Moreover, the  $\text{Si}_3\text{N}_4$  materials with the addition of  $\text{Lu}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  have better high-temperature properties compared with ceramics containing  $\text{Sm}_2\text{O}_3$  or  $\text{La}_2\text{O}_3$ . Then, during the “ball-on-disc” tests, the tested sample or the standard  $\text{Si}_3\text{N}_4$  ball could get heated to higher temperatures, because of the lack of lubricant, and this exhibits the high-temperature characteristics of the material.

## 10. Use of carbon nanotubes as reinforcement material

Recently, CNTs are considered for use as reinforcements in advanced composite materials in view of their high elastic modulus and strength compared to existing fibers. In that respect, being rolled up graphite sheets, their mechanical properties are expected to be equal to or greater than the value for a graphene sheet. Qualitative and quantitative TEM and AFM studies have been performed on individual tubes and have confirmed their superior strength and stiffness. The Young's modulus of a CNT is over 1 TPa and its estimated tensile strength is over 150 GPa, i.e. they are a hundred times stronger than steel, though three to five times lighter.

The properties of carbon nanotubes are a mix of diamond and graphite: strong, thermally conductive like diamond; electrically conductive like graphite; and are light and flexible. Many potential applications have been proposed for carbon nanotubes like: conductive polymers; energy storage and energy conversion devices; sensors; field emission displays; replacing silicon in microcircuits; multilevel chips; probes for SPM (scanning probe microscopy).

The objective of current investigations is to prepare, by sintering in the presence of liquid phase, a dense ceramic material based on SiC with sintering additives, with the addition of CNTs, resulting in a composite with high electrical conductivity, while maintaining its good mechanical properties.

### 10.1 Sintering in the presence of liquid phase

This type of sintering is one of the used techniques to accelerate the process, while maintaining the mechanical properties of the material. The liquid phase is understood as an additive which is liquid at the sintering temperature. This liquid phase must fulfill two basic conditions: first, it has to drench all of the areas of the solid phase, and second, the sintered material has to have the liquid phase grains partly dissolved.

Sintering in the presence of liquid phase consists in three processes. First, we have the formation of the liquid phase and its flow, which causes changes in the spatial organization of the grains at the solid formation. This process is called relocation and it often leads to a sharp increase on the density of the sintered material. The relocation, which is due to the capillary forces of the liquid phase wetting the solid grains, is dependent on the amount of liquid phase, its viscosity, and the grain size and solubility of the solids in the liquid. The next and most important process for sintering in the presence of liquid phase is the process of increasing the density by dissolving the solid grains in the liquid phase and then the process of the subsequent elimination.

It is thus clear, that the melt should have the lowest possible viscosity at the sintering temperature and the highest possible at the application temperatures. These are conflicting requirements, and are very difficult to joint. The chemical composition of the phases at the grain boundaries is the subject of study of many authors, because grain boundaries have a significant effect on the mechanical and functional properties of the material.

### 10.2 Formation of the CNTs by CCVD

The CNTs can be added by a method called catalytical chemical vapor deposition (CCVD), where tubular carbon nanofibers are formed on a support by a catalytical deposition of carbon out of the vapor phase. This method is characterized by a catalyst layer, which is deposited without current, and is applied to the support at least in places. Said catalyst layer is thermally activated before depositing carbon out of the vapor phase in a reducing atmosphere. Since it was discovered, that the elements of the iron group, Fe, Co and Ni, are catalytically active, these elements are used as catalytics.

## 11. Conclusion

In the present paper are discussed issues of ceramic materials as basic elements of composite materials, which present advanced technical materials for a wide practical use. The attention was focused mainly on the causes of brittleness on ceramics and the possibilities of its

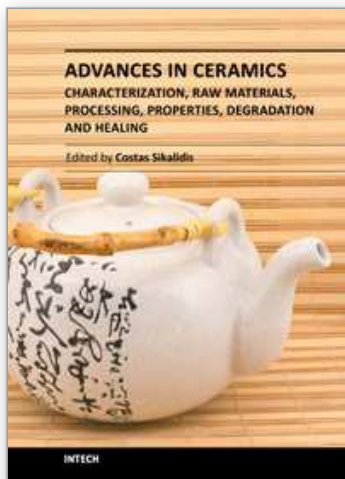
elimination. There were also discussed issues of composite materials based on ceramics considering their preparation and evaluation of selected properties. The results obtained extend the knowledge about the preparation of the above mentioned advanced materials with the addition of rare earth metals and their selected mechanical properties.

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## **Advances in Ceramics - Characterization, Raw Materials, Processing, Properties, Degradation and Healing**

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The current book consists of eighteen chapters divided into three sections. Section I includes nine topics in characterization techniques and evaluation of advanced ceramics dealing with newly developed photothermal, ultrasonic and ion sputtering techniques, the neutron irradiation and the properties of ceramics, the existence of a polytypic multi-structured boron carbide, the oxygen isotope exchange between gases and nanoscale oxides and the evaluation of perovskite structures ceramics for sensors and ultrasonic applications. Section II includes six topics in raw materials, processes and mechanical and other properties of conventional and advanced ceramic materials, dealing with the evaluation of local raw materials and various types and forms of wastes for ceramics production, the effect of production parameters on ceramic properties, the evaluation of dental ceramics through application parameters and the reinforcement of ceramics by fibers. Section III, includes three topics in degradation, aging and healing of ceramic materials, dealing with the effect of granite waste addition on artificial and natural degradation bricks, the effect of aging, micro-voids, and self-healing on mechanical properties of glass ceramics and the crack-healing ability of structural ceramics.

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