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Toughening and strengthening of advanced ceramics with rare earth additives

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Abstract

Al_2O_3/(W,Ti)C composite ceramic materials reinforced by rare earth additives have been fabricated by the hot pressing technique. Microstructure, flexural strength, toughening and strengthening mechanisms are analyzed. The maximum flexural strength and fracture toughness of the yttrium reinforced Al_2O_3/(W,Ti)C ceramic are 853 MPa and 6.1 $MPam^{1/2}$, respectively, which are approximately 20% and 16% higher than for the corresponding material without yttrium. Acting mechanisms of the yttrium additive are mainly the purification of interface, the subsequent increase in the interface bonding strength and strengthening by nanometer sized particles. Because of the co-existence of both strong and weak interfaces, synergistic interactions of toughening mechanisms like crack bridging, crack branching, crack defection and microcracking, etc. are enhanced. Therefore, fracture toughness of the rare earth reinforced Al_2O_3/(W,Ti)C ceramic material is increased. The developed new ceramic materials are found to achieve higher fracture resistance when used as cutting tools.

Keywords: C. Mechanical properties; C. Toughness and toughening; D. Al_2O_3; E. Cutting tools

1. Introduction

At present, Al_2O_3-based ceramic material is one of the most widely used ceramics in practice. However, the intrinsic brittleness still exists as a fatal weakness for ceramic materials. In order to reduce the brittleness and to increase the strength and toughness, a great deal of research work has been done [1–3]. Mechanical properties of alumina-based ceramics have been improved by the incorporation of one or more reinforcing phases such as TiC, TiN, TiB_2, SiC particulates, SiC whiskers, B_4C, ZrO_2, WC, (W,Ti)C, Ti(C,N), Cr_3C_2, NbC, etc. Investigations on toughening mechanisms such as crack deflection, crack branching, crack bridging, microcracking, transformation, toughening by residual thermal stress and their synergistic interactions, etc. are still active [1,4–8].

Rare earth elements, working as a series of effective additives, have got widespread applications in current research of advanced ceramic materials [9,10]. They can be used not only as the stabilizer of the tetragonal zirconium oxide but as the sintering aid for Al_2O_3, TiB_2, TiC, SiC, Si_3N_4, sialon and AlN ceramics [9–12]. As a result, physical and mechanical properties of these ceramics have been noticeably enhanced. However, other possible acting mechanisms of rare earth elements have rarely been reported elsewhere. In the present study, Al_2O_3/(W,Ti)C ceramic composites reinforced by rare earth additives are fabricated. Microstructure, mechanical property, toughening and strengthening mechanisms and cutting performance are investigated in detail.

2. Experimental procedures

Commercially available high purity alumina and (W,Ti)C powders were used as the starting materials with average sizes of 0.8 μm and 1.0 μm, respectively. The raw materials were blended with each other (with 35 wt.% (W,Ti)C) and doped with different amounts up to 2 wt.% of pure rare earth

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metal additive such as yttrium (Y). The doping process and the following ball-milling process have been performed under a N_2 protective atmosphere. The mixtures were subsequently homogenized with absolute alcohol media in a ball mill for 120 h. After milling, the slurry was dried in vacuum and screened under N_2 atmosphere. Samples were then hot pressed in a N_2 atmosphere in a graphite mould at 1740 °C, holding time of 30 min and at 35 MPa. Sintered bodies were then cut with a diamond wheel into specimens and inserts.

The three point bending method was used to measure the flexural strength with a span of 20 mm and a cross head speed of 0.5 mm/min on 3 mm × 4 mm × 40 mm test bars carefully ground and polished to an average surface roughness less than 0.1 μm. The fracture toughness was determined on bars of the above size by a four point SENB (single edge notched beam) method with a 10 mm inner span and a 20 mm outer span at a cross head speed of 0.1 mm/min. Hardness was measured with a Vickers hardness tester (model HV-120) with a static load of 196 N and a holding time of 15 s. Data for flexural strength, fracture toughness and hardness were gathered on five specimens for each run.

Microstructural characterizations of the material were performed with scanning electron microscope (SEM, model HITACHI S-570) and transmission electron microscope (TEM, model HITACHI H-800) equipped with the energy spectrum analyzer and operated at 175 kV. Samples used for the TEM analysis were first ground and polished mechanically to be less than 50 μm, and then ion thinned to perforation.

The developed ceramic material was then utilized as a tool material, and cutting tests were done according to the following cutting conditions. Intermittent machining was performed on a CA6140 lathe equipped with a 75° lead angle, 5° negative inclination, 5° negative rake tool holder and 5° clearance. The geometrical parameter of tool inserts is SNGN160604 with an edge chamfer of 0.2 mm at 20°. The work material is a 0.45% C mild carbon steel (#45 steel) with a hardness of 46–48HRC in the form of round bars with four symmetrically distributed longitudinal slots of 8 mm in width and 40 mm in depth. Tool materials used for comparison are WT1, WT2 and WT0 which corresponds to Al_2O_3/35 wt.% (W,Ti)C ceramic materials with 0.5 wt.% Y, 2 wt.% Y and without yttrium, respectively.

3. Results and discussions

3.1. Mechanical property

The maximum flexural strength of Al_2O_3/(W,Ti)C ceramics is about 853 MPa when 0.25 wt.% Y is added (Fig. 1), which is about 20% higher than that of the corresponding material without yttrium. Then, with the increase of yttrium content, the flexural strength decreases slowly.

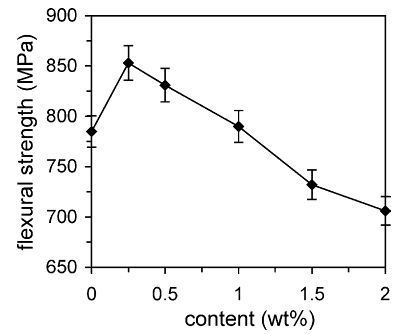

Fig. 1. Effect of yttrium on the flexural strength of Al_2O_3/(W,Ti)C ceramics.

Under the experimental conditions, the incorporation of yttrium can improve the fracture toughness of Al_2O_3/(W,Ti)C ceramic materials when its content is less than 1.5 wt.% (Fig. 2). Particularly, when 0.5 wt.% Y is added the fracture toughness reaches 6.1 $MPam^{1/2}$, which is approximately 16% higher than that of the corresponding ceramic without yttrium. However, the addition of yttrium resulted in a general slight decrease in the hardness (Fig. 3).

3.2. Microstructure

The microstructure of rare earth reinforced Al_2O_3/(W,Ti)C ceramic materials are affected by the amount of rare earth additives. As can be seen in Fig. 4, the microstructure of 0.25 wt.% Y ceramic is nearly the same

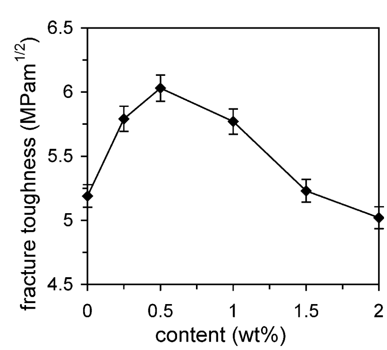

Fig. 2. Effect of yttrium on the fracture toughness of Al_2O_3/(W,Ti)C ceramics.

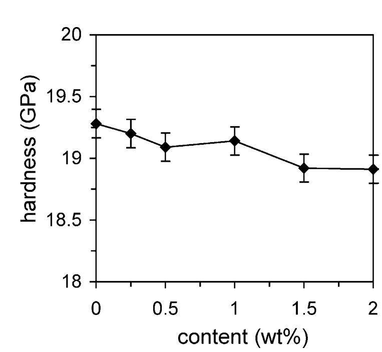

Fig. 3. Effect of yttrium on the hardness of Al_2O_3/(W,Ti)C ceramics.

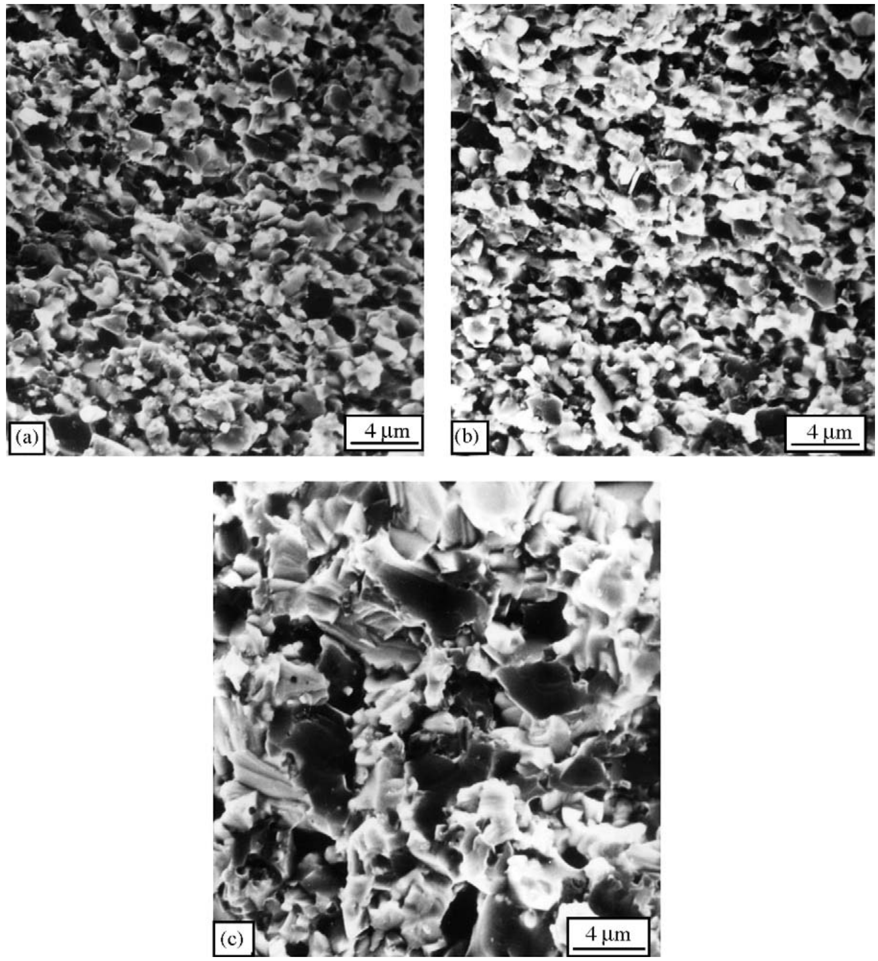

Fig. 4. Effects of rare earth element on the microstructure of Al_2O_3/(W,Ti)C ceramics: (a) without yttrium; (b) with 0.25 wt.% Y; (c) with 2 wt.% Y.

as that of the corresponding material without yttrium (Fig. 4 (a) and (b)). The (W,Ti)C phase is distributed homogeneously with grain sizes of 1–2 μm in the Al_2O_3 matrix. Systematic experiments show that the incorporation of less than 0.5 wt.% yttrium has little effect on the microstructure of Al_2O_3/(W,Ti)C ceramic material. When the yttrium content is 2 wt.%, most of grains of the material are larger than 2 μm in size and some even reach 6–7 μm (Fig. 4 (c)). It suggests that the excessive addition of yttrium will result in the abnormal grain growth and inhomogeneous distribution of grains and the decrease of the flexural strength which is only 706 MPa (Fig. 1). Thus, only when the addition of yttrium below a certain amount can the deteriorate effect on the microstructure of Al_2O_3/(W,Ti)C ceramic material not be found.

Fig. 5 gives the microstructural characteristics of Al_2O_3/(W,Ti)C ceramic material with yttrium addition. Some black spherical or particulate-like compounds with grain sizes of 20–100 nm are found to exist inside the alumina grains in addition to the main phases of Al_2O_3 and (W,Ti)C in the 0.5 wt.% Y ceramic material (Fig. 5(a)). Results of the energy spectrum analysis of compound A and compounds B and C in Fig. 5(b) and (c), respectively are all given in Table 1. The elements Fe, Ni, Cr and part of W in Table 1 are all impurities induced in ball-milling process. It indicates that there exist some yttrium and impurity elements including W and Ni in the black compounds (Fig. 5(a)), which might be the yttrium containing complex compounds. As a result of the existence of these nanometer sized particles, the alumina matrix can be reinforced with the

Table 1
Results of the energy spectrum analysis (wt.%)

Sample number	Analysis area	Al	Ti	Ni	Y	W	Fe	Cr
(a)	A	59.41	18.73	1.46	0.98	19.42	–	–
(b)	B	42.38	20.84	3.06	1.87	21.68	8.92	1.25
(c)	C	38.51	25.97	6.64	1.63	4.07	22.34	0.84
(d)	D	51.65	23.11	–	–	25.24	–	–

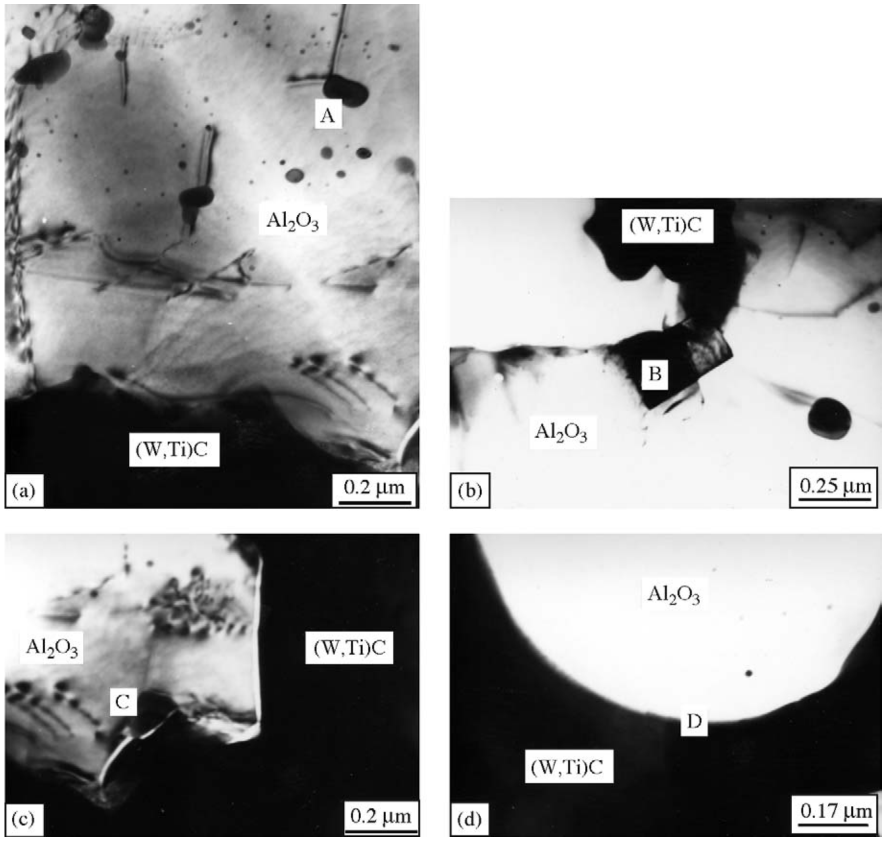

Fig. 5. TEM morphology of the microstructure of Al_2O_3/(W,Ti)C ceramics with 0.5 wt.% Y: (a–c) yttrium containing compounds; (d) clean interface of Al_2O_3–(W,Ti)C.

similar toughening and strengthening mechanisms as in nano-ceramics. Additionally, in the cases of Fig. 5 (b) and (c) the amount of impurity elements Fe, Cr, Ni and W is relatively high which implies that yttrium may react with one or several elements among these impurities and work to gather the impurity elements. Thus, interfaces are purified by the reactions, and the binding strength and the resistance to crack propagation are enhanced. As a result, both flexural strength and fracture toughness are improved.

The typical morphology of the clean interface of Al_2O_3–(W,Ti)C is given in Fig. 5 (d). Results of the spectrum analysis of the interface area (analysis area D in Fig. 5 (d)) also prove it to be clean where no impurities can be detected except Al, Ti and W elements (sample number (d) in Table 1). The kind of clean interfaces can work as strong interfaces, while those where impurity containing rare earth compounds exist become weak interfaces. Both strong and weak interfaces exist concurrently in the yttrium reinforced Al_2O_3/(W,Ti)C ceramic materials.

3.3. Toughening and strengthening mechanism

Indentation crack propagation patterns could be, to some extent, regarded as the microstructural characterization of toughening mechanisms of ceramic materials. In Fig. 6, the indentation crack in Al_2O_3/(W,Ti)C material without yttrium is relatively wide and straight and no obvious crack deflection, crack bridging, crack branching and their interactions happen.

But for the yttrium reinforced ceramics, morphologies of indentation cracks are completely different (Fig. 7). In Fig. 7(a), the main crack is branched near point A at first,

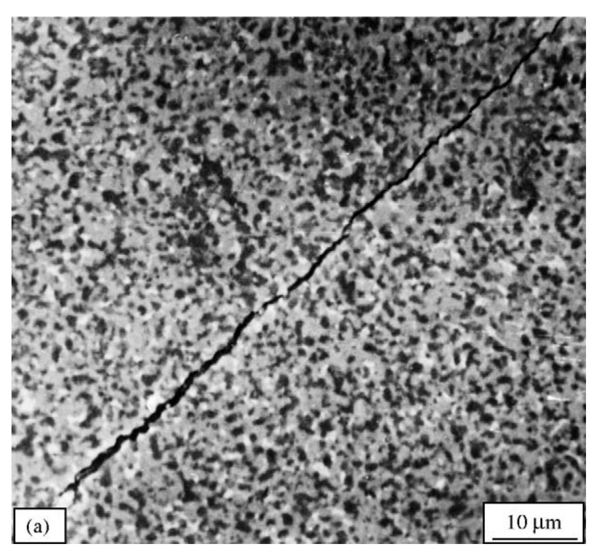

Fig. 6. Indentation crack in Al_2O_3/(W,Ti)C ceramics without yttrium under SEM.

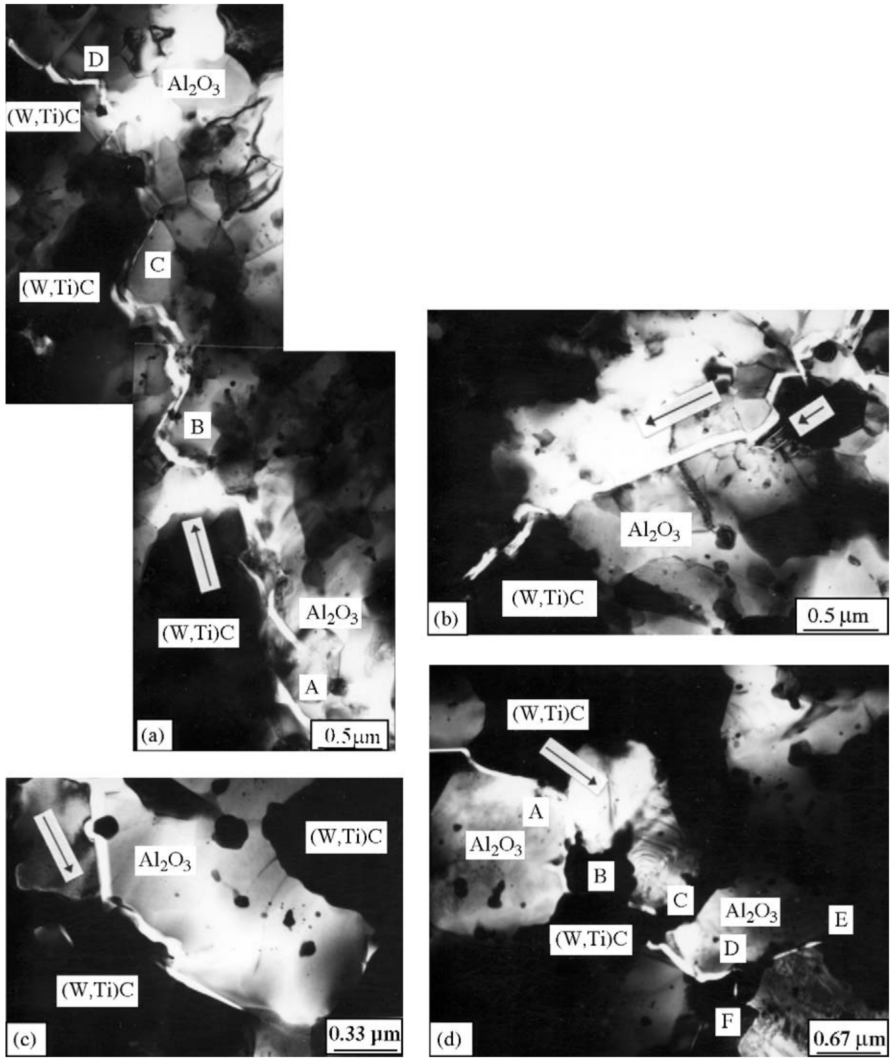

Fig. 7. Morphology of the crack propagation in 0.5 wt.% Y reinforced Al_2O_3/(W,Ti)C ceramics under TEM.

then deflected by grain B. When the crack extends into the interface of Al_2O_3–(W,Ti)C, it becomes narrower quickly and finally stopped (point C). A new secondary crack originates from the interface area and continues to extend forward (point D). In Fig. 7(b), when the crack propagates near the phase boundary of Al_2O_3–(W,Ti)C, spontaneous microcracks are firstly formed under the action of residual thermal stresses. Then, the crack penetrates through several Al_2O_3 grains and eventually is pinned and stopped inside a (W,Ti)C grain. The phenomenon is nearly the same as that observed by Li. et al. [13] when the ceramic sample is tensioned dynamically inside the high voltage electron microscopy. The crack in Fig. 7(c) is firstly affected by the pulling-out of a small (W,Ti)C grain. Then, it is deflected and inhibited and finally stopped at the phase boundary of Al_2O_3–(W,Ti)C. In Fig. 7(d), the extending crack undertakes the deflection at point A, bridging at points B and C by two (W,Ti)C grains and later the deflection again and crack branching at point D. After that, the crack branches become smaller and disappear inside a (W,Ti)C grain E and at the boundary area of Al_2O_3–(W,Ti)C (point F). These kinds of phenomena can easily and popularly be observed in the developed yttrium reinforced Al_2O_3/(W,Ti)C ceramic materials.

In fact, the purification of most of the interfaces means the increase of interfacial bonding strength, which may cause the inhibition of cracks at the interfaces [14]. On the contrary, interfaces where rare earth compounds exist become weak. The interfacial bonding strength is consequently decreased which makes it prone for the cracks to extend along the interfaces. Since the particles of rare earth compounds are extremely small, most of which are less than 100 nm, and only a minute amount of yttrium is incorporated into the material, the resulted weak interfaces are just in a small quantity. Thus, no obvious weakening to the material properties can be observed. However, with the increase of yttrium content, the ratio between strong and weak interfaces changes slowly. Strong interfaces become less and weak interfaces become more. Because of the co-existence of both strong and weak interfaces, toughening mechanisms like crack bridging, crack

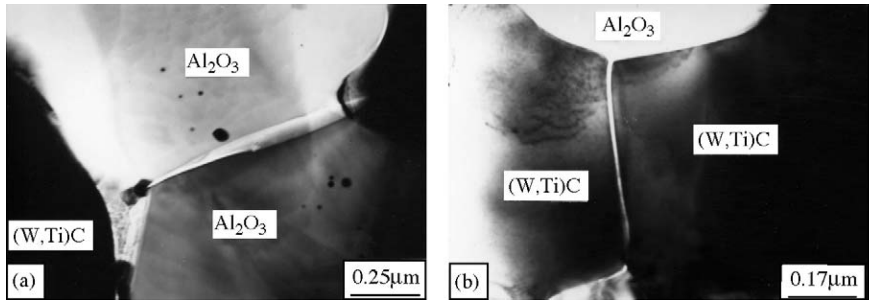

Fig. 8. TEM morphology of spontaneous cracks in 0.5 wt.% Y reinforced Al_2O_3/(W,Ti)C ceramics.

branching, crack defection, microcracking and their synergistic interactions are noticeably intensified (Fig. 7). It can reasonably be concluded that fracture toughness of rare earth reinforced Al_2O_3/(W,Ti)C ceramic material is increased as a result of the synergistic interactions among these toughening mechanisms.

Other TEM morphologies of spontaneous microcracks in yttrium reinforced Al_2O_3/(W,Ti)C ceramic are shown supplementally in Fig. 8. As can be seen, spontaneous microcracks exist not only at the phase boundaries of Al_2O_3–(W,Ti)C but at the grain boundaries of Al_2O_3–Al_2O_3 or (W,Ti)C–(W,Ti)C. According to the literature [15], residual thermal stress will be formed as a result of the mismatches in the coefficient of thermal expansion and Young's modulus. They usually result in the microcracks along the boundaries. The formation of microcracks will undoubtedly cause branching or torsion of the main crack along the way it propagates. Furthermore, analyses by Swanson et al. [16] have revealed that crack branching usually happens together with other single toughening mechanisms such as crack defection and microcracking instead of happening individually. While crack bridging is always accompanied with the crack deflection in whisker reinforced ceramics [17]. The work here gives a further experimental verification on this point.

3.4. Cutting performance

Higher fracture resistance is found for the yttrium reinforced Al_2O_3/(W,Ti)C ceramic material WT1 in intermittent machining of hardened carbon steels when used as cutting tools (Fig. 9). The impact number in Fig. 9 indicates the number of intermittent cuts that can be sustained prior to fracture. The fracture resistance of WT1 is 17–21% higher than that of WT0 and 27–36% higher than that of WT2 under three groups of cutting conditions (group A: cutting speed $v = 118$ m/min, feed rate $f = 0.1$ mm/rev, depth of cut $a_p = 0.5$ mm; group B: $v = 188$ m/min, $f = 0.1$ mm/rev, $a_p = 0.3$ mm; group C: $v = 264$ m/min, $f = 0.1$ mm/rev, $a_p = 0.1$ mm). It seems that fracture resistance of Al_2O_3/(W,Ti)C ceramic material can noticeably be improved as a result of the proper addition of yttrium additives. The developed ceramic material will have certain advantage in the field of intermittent machining of hardened carbon steels.

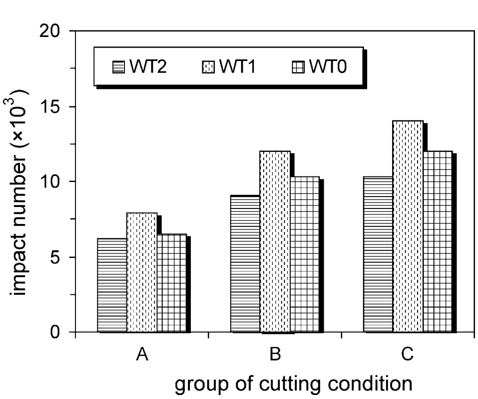

Fig. 9. Fracture resistance of Al_2O_3/(W,Ti)C series ceramic materials.

4. Conclusions

The addition of rare earth additives such as yttrium of a suitable amount in a proper way can notably improve the flexural strength and fracture toughness and fracture resistance of Al_2O_3/(W,Ti)C ceramic material.

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